3347

ChemRisk/Shonka Research Associates, Inc., Document Request Form

(This section to be completed by subcontractor requesting document)

Y. Moon / OSTI

Requestor / Document Center (is requested to provide the following document)

Date of request 4/4/97 Expected receipt of document ______

Document number C-2.16.13 (SRD) / K/EM-478 Unclassified Date of document 4/23/97

Title and author (if document is unnumbered)

Sanitized Version of Accounting for T and X in the Diffusion Plant Dated 4/3/46

(This section to be completed by Document Center)

Date request received 4/7/97

Date submitted to ADC 4/7/97

Date submitted to HSA Coordinator 4/7/97

(This section to be completed by HSA Coordinator)

Date submitted to CICO (Sanitize) 4/7/97 4/23/97 (Release)

Date received from CICO 4/23/97 4/28/97

Date submitted to ChemRisk/Shonka and DOE 4/29/97

(This section to be completed by ChemRisk/Shonka Research Associates, Inc.)

Date document received ______

Signature ______

Sheila Thornton
4-9525
K-303-8 MS 7314

3347

6605

w/3 attachments
This document consists of 176 pages
No. 2 of 2 copies, Series B
(1870d)

Cook	
Archer	
Irvine	
Luke	OK
Baranowski	OK
Anderson	OK
Rogers	OK
Peyton	
Mail & Records	OK

CARBIDE AND CARBON CHEMICALS CORPORATION

COPY
FORWARDED BY
C. E. CENTER

PROCESS DIVISION

PROCESS DEVELOPMENT DEPARTMENT

April 3, 1946

To: Mr. C. N. Rucker, Jr.

ACCOUNTING FOR T AND X IN THE DIFFUSION PLANT

The attached report of this title by Mr. A. M. Squires, Mr. C. Daniel and the writer, is the first step towards setting up a reliable material accounting system for the K-25 plant.

The report presents an analysis of the problem from the point of view of the Process Development Department, to serve as a basis for discussion with the operating departments and the laboratory.

M. Benedict
M. Benedict

DISTRIBUTION:

S. Barnett	A. B. Hale	W. C. Moore
Walter Beard	A. P. Huber	J. Newman
M. Benedict	D. E. Hull	H. Nowak
C. E. Center	F. W. Hurd	H. F. Priest
R. Conner	M. F. McDermott	O. Rinehart
J. Connors	D. A. MacRae	C. N. Rucker, Jr.
C. Daniel	J. B. Marcum	G. T.E. Sheldon
L. Davidson	J. A. Marshall	A. M. Squires
G. A. Garrett	E. E. Minett	J. L. Waters

Major C. Luke
Eng. Fac.

DEPARTMENT OF ENERGY DECLASSIFICATION REVIEW

1st Review - Date: 4-25-97	Determination [Circle Number(s)]
Authority: ☐ADC ☒ADD	1. Classification Retained
Name: mThem	2. Classification Changed To:
2nd Review - Date: 4/25/97	3. Contains No DOE Classified Information
Authority: ADD	4. Coordinate With:
Name: George Holmes	5. Classification Cancelled *
	6. Classified Information Bracketted
	7. Other (Specify):

Su B

DOE-OR QA gm
~~CHANGED~~ 4/29/97 ~~SECRET~~

~~This document contains information affecting the National Defense of the United States within the meaning of the Espionage Act, U.S.C. 50: 31 and 32. Its transmission or the revelation of its contents in any manner to an unauthorized person is prohibited by law.~~

* This Extract Abstract
Pages i - V
" 1-10
Table 1-1A
Figures 1-4 *

RHTG # 104839
BOX # 228

2.16.13

[redacted]

~~RESTRICTED DATA~~

~~This document contains Restricted Data as defined in the Atomic Energy Act of 1954. Unauthorized disclosure subject to Administrative and Criminal Sanctions.~~

CARBIDE AND CARBON CHEMICALS CORPORATION

PROCESS DIVISION

PROCESS DEVELOPMENT DEPARTMENT

Report No. C-2-16-13

Date: April 3, 1946

TO: C. N. Rucker, Jr.

Written by: M. Benedict

C. Daniel

A. M. Squires

ACCOUNTING FOR T AND X IN THE DIFFUSION PLANT

ABSTRACT: It is recommended that a reliable material accounting system be set up at the diffusion plant, so that material balances for T and X may be evaluated with the least practicable uncertainty. Suggestions are made for systematizing material accounting. Specific procedures are recommended for most of the transfers of material and taking of inventories, subject to further study by the Process and Laboratory Departments. Additional technical studies are recommended to improve the material accounting system.

An analysis is made of the reliability of material balances at K-25. It is recommended that a material balance period be about 30 days in length. In such a period the daily rate of loss of T can be determined with an uncertainty of about $\pm$1.2 kgm. and the daily rate of loss of X with an uncertainty, under present assay techniques, of about $\pm$0.1 kgm. The latter number may be reduced to about $\pm$0.04 kgm. by further research on assay methods. Both the T and X uncertainties are further reduced when the data from a number of material balance periods are pooled.

A material accounting system of this degree of reliability will furnish valuable information concerning the rate of consumption of TF_6 by plant surfaces and concerning the possibility of unreported diversion of X from the K-25 area.

TABLE OF CONTENTS

LIST OF TABLES

LIST OF FIGURES

[redacted]

ACCOUNTING FOR T AND X IN THE DIFFUSION PLANT

I. INTRODUCTION

1. Importance of the Problem.

The extreme monetary and strategic value of the X in process in the K-25 and K-27 Plants demands that an intensive effort be made to set up accounting methods for the T and X handled by these plants that are as precise as possible. The validity of judgments of the performance of the plants during a given period is limited by the reliability of the accounting procedures used. Judgments of the possibility that X may have been illegally diverted from the K-25 area during a given period are limited by the precision with which the unaccounted losses of X during the period may be determined, the records of the amount of unaccounted loss of X in earlier comparable periods, and the degree to which these losses can be certified to be legitimate (i.e., corrosion of metals by UF_6, escaped purge gases, spillage, etc.)

During the war months, when it was important to rush the plant into service, the refinements in operating techniques which are required for accurate material accounting were temporarily disregarded. Now that the plant is on a peace-time basis, with emphasis on economy of operation, the establishment of the most

[redacted]

[redacted]

reliable practicable material accounting system is a primary operating responsibility.

2. Purpose of Report

This report makes the first necessary step toward establishing such a material accounting system. The problems met in accounting for T and X in the diffusion plant are analyzed and provisional material accounting procedures are outlined. As subsequent steps in establishing a material accounting system, the operating departments should draw up detailed accounting procedures based on the provisional procedures of this report and should modify these procedures if proved desirable by practical experience with them, in accordance with the principles of this report.

Because of the complexity of the K-25 plant, the variety of forms in which T and X are handled, and the large inventory of T and X in comparison with the daily product rate, reliable accounting for these materials is an unusually difficult undertaking. Nevertheless, this report demonstrates that such accounting is a feasible undertaking and outlines the principles and general procedures which should be followed if a satisfactory material accounting system is to be developed.

3. Scope and Organization of Report

Section II summarizes the principal conclusions of this report concerning the precision possible in material accounting, the procedures recommended for this purpose and the frequency with which it is desirable that inventories and material balances be made.

[redacted]

[redacted]

Section III lists the principal recommendations of this report concerning the nature and organization of future work on material accounting.

The remaining four sections of the report give the detailed information on which Sections II and III are based. Section IV describes the flow of T and X in the diffusion plant and proposes a flow sheet and terminology for describing all operations in which T and X are stored and all transfers of these materials. This Section answers the questions:

(1) How should sections of the plant be grouped or broken down for the purpose of taking inventory of T and X?

(2) At what points in the plant should accurate measurements be made of the flow of T and X from one section or department to another?

Section V answers the question:

(3) For what groups of operation should material balances be evaluated?

Section VI explains how a material balance on the main process can be used to provide some information concerning the rate of consumption of process gas in the plant and lists additional information which is needed for a reliable evaluction of the consumption rate. The likelihood of illegal diversion of X from the K-25 area may be judged on the basis of all of this information concerning consumption taken together.

Section VII gives a detailed analysis of the precision and accuracy attainable in the most important material balance,

[redacted]

[redacted]

that on the main process. Answers are given to the following questions:

(4) If various degrees of precision and accuracy of measurment of flows, pressures, assays, etc. are assumed, what uncertainty results in the main process material balance?

(5) What additional work should be done to decrease the uncertainty in the material balance?

The answer to question (4) together with the best current guess of the ultimate reliability with which data can be obtained for a material balance, provides a tentative estimate of the uncertainty to be expected of the final material balance procedure. The answer to question (5) indicates where future effort should be concentrated to reduce the error in material balances.

It has not been possible to make a complete study of the reliability of all measurements entering the material balance, nor has it been possible to review all operating procedures which affect material accounting, with a view to improving their reliability. A program is outlined, in Section III, for extending the methods of this report to an exhaustive analysis of all procedures and data. This analysis will require the services of several competent engineers and should preferably be made by those finally to have responsibility for the material balance, in cooperation with the Process Analysis Section. After the completion of this work and the carrying out of several material balances, a better estimate of the reliability of the material balance will be possible.

[redacted]

[redacted]

Section VIII recommends certain general features desirable in any material accounting system, which have become apparent from the study of this report. In addition, partial recommendations are given concerning the specific procedures to be followed in some of the individual measurements required in material accounting. These recommendations are made as a guide to the group who will have the responsibility of drawing up the detailed material accounting procedure.

[redacted]

[redacted]

II CONCLUSIONS

1- The material flow sheet, Figure A, bound in the rear of this report, shows the pattern proposed for describing the storage and transfer of T and X in the diffusion plant. The rectangles of this flow sheet represent the operations to which it is proposed all T and X inventory be assigned. The lines connecting rectangles represent the routes by which it is proposed that all transfers of T and X be described. The terminology and numbering code of this flow sheet are used consistently throughout the report.

2- Material balances may be advantageously computed for six portions of the plant:

(1) Overall Material Balance for entire plant

(2) Main process material balance

(3) Laboratory material balance

(4) Conversion department material balance

(5) Coded chemicals material balance

(6) Spent carbon and alumina material balance.

The boundary of the process area material balance, the most important area, is indicated by the heavy, full line of Figure A.

3- Possible diversion of X from the main process and consumption of TF_6 by plant surfaces may be partially evaluated from unaccounted losses of the main process material balance by the following equation:

Diversion + Consumption = "Unexplained Losses" =

Unaccounted Losses + X recovered from Process Equipment.

[redacted]

The amount of consumption alone can be roughly estimated from laboratory measurements of consumption rate or from the records of the Decontamination and Recovery Department. A more accurate estimate can be obtained by removing all the equipment of several normal cells and making a careful analysis of the T deposited in it. The relative amounts of diversion and consumption can be estimated from the ratio of the unexplained loss of X to the unexplained loss of T.

4- An analysis is made of the reliability of material balances on T and X in the main process area of the K-25 plant alone. The analysis is indicative of the reliability for the combined K-25 and K-27 plants, and may be readily extended to them. Table I lists assumptions made concerning the number of repeated measurements and the precision or accuracy of individual measurements of the more important quantities entering the main process material balance.

5- From this analysis, it is concluded that the change in T inventory from the beginning of a material balance period to the end is 95% certain to lie within ±34 kgm. of the correct value, and the change in X-inventory within ±0.9 kgm. of the correct value, even though individual inventories cannot be determined with this accuracy. The inventory of sections 300 and 600 is about 10,000 kgm. T and 200 kgm X. These uncertainties in the change in inventory set a lower limit to the uncertainty of the material balance, even when there is no uncertainty in the

[redacted]

[redacted]

measurement of the amount of T and X transferred in feed, product and waste streams.

6- When both random and systematic errors in the measurement of amounts of T in feed, product and waste streams are taken into account, the uncertainty in the total loss of T will depend on the duration of the material balance period, as illustrated in Figure 1. For periods of time under 30 days, most of the uncertainty is due to uncertainty in inventory. For longer periods of time the uncertainty in T-streams is also a factor. Figure 2 illustrates the uncertainty in the daily rate of loss of T evaluated from the T-material balance. It is not possible at present to give a reliable estimate of the daily rate of loss of T to be expected in material balances. It is believed that the loss is not less than 2 kgm. T/day, or greater than 20 kgm. T/day. If the higher rate is found, it can be evaluated within 10% by using material balance periods longer than 18 days. The uncertainty in daily rate of loss of T can be made practically negligible after material accounting has been in effect longer than one year.

7- The uncertainty in the rate of loss of X cannot be made negligible by extending the period of material accounting, because the X assay of feed, product and waste will probably be affected by small systematic errors, or biases. Figure 3 illustrates the uncertainty in the rate of loss of X for various assumptions concerning bias in assays and various material balance periods. With present estimated biases, the uncertainty in rate of loss of X shown in Curve I, cannot be reduced to less than ± 0.085 kgm. X/day,

[redacted]

and there is little advantage in extending the material balance period beyond 30 days. If the bias in X-essay is reduced to an extent believed attainable after a moderate amount of additional research, the uncertainty in rate of loss of X, shown in Curve III, can be reduced to $\pm$ 0.02 kgm. X/day, by using a material balance period of 100 days. The advantage to be gained in reducing the estimated present bias in X-assay is clear. It is believed that the daily rate of loss of X is not less than 0.04 kgm.X/day, or greater than 0.40 kgm.X/day. If a value toward the upper end of this range is found, it will be important to investigate immediately the possibility of unreported diversions.

8- Figure 4 illustrates the uncertainty in the total loss of X for various assumptions concerning bias and various material balance periods. After one year, the uncertainty in the unaccounted loss of X would be 31 kgm. for the estimated present biases and 6 kgm. for the biases believed attainable.

9- Figures 1, 2, 3 and 4 may be used to set a preferred duration of material balance period. If material balance periods are too short, uncertainties in inventory will make the daily rates of loss very uncertain. If the periods are too long, biases in assay may obscure large losses that occurred at one particular time which could have been found by more frequent material balances. The best procedure appears to be to use a material balance period a little shorter than one which will give the desired certainty, and if the results for single periods are too irregular, report results only for two or more single periods evaluated as one longer period. As

[redacted]

an arbitrary rule, it is suggested that material balances be evaluated with such frequency that the uncertainty in the unaccounted rate of loss of X for a single period is twice the uncertainty attainable from a large number of periods. Using this criterion, material balances should be evaluated every 30 days if the bias in X-assay can be reduced to the extent now believed attainable. Until the bias in X-assay can be reduced, a shorter period, such as 7 or 14 days, could be used with some advantage, except for the difficulty of taking complete inventories so frequently. Since it would be difficult to carry out a complete inventory of the entire plant this frequently, it is recommended that a material balance period of 4 weeks, or one month, be adopted initially. Experience with the first balances evaluated for this period will indicate whether the period should be shortened or lengthened. It is felt that the material balance period should not be extended greatly beyond thirty days because of the risk of accidental spillage or other mishap which may vitiate the data obtained.

[redacted]

[redacted]

TABLE I, Part (a)

Summary of Assumptions Underlying Conclusions on Reliability of Material Balances

Measurements of Inventories

Quantity Measured	Number of Measurements per Inventory	Precision or Accuracy (95% Confidence Belt on Single Measurement, except where noted)
Cell Calibration Size 1	0	$\pm$3% on average*
Size 2	0	$\pm$3% on average*
Size 3	0	$\pm$3% on average*
Size 4	0	$\pm$5% on average*
Number of Cells Onstream	1	$\pm$0
Stage Tails Pressures**	2900	$\pm$0.1 psia
Stage Control Valve Angle	2900	$\pm$5% closed
Stage Temperature	2900	$\pm 5^{\circ}$ F
Cell % N_2	100	$\pm$10%
Cell %816	36	$\pm$10%
Intercell Piping Pressure	475	$\pm$0.1 psia
Inter-building Piping Pressure	51	$\pm$0.1 psia
Section 600 Surge Drum Pressure	1	$\pm$0.05 psia
Section 600 Surge Drum Temperature	1	$\pm 5^{\circ}$F
Section 312 Stage Pressure	40	$\pm$0.1 psia
Building X-Assay (precision)	2 per building	$\pm$1.8%
Building X-Assay (accuracy)	--	1.0%

*This is confidence belt on cell calibration used in calculating inventory, this being the average of several calibration tests.

** It is assumed that the change in tails pressures between two inventory times does not exceed 10%.

[redacted]

[redacted]

TABLE I, Part (b)

Summary of Assumptions Underlying Conclusions on Reliability of Material Balances

Measurements of Transfers

Quantity Measured	Number of Measurements per Drum	Precision or Accuracy (95% Confidence Belt on Single Measurement)
Feed Drum Weight	2(full) 2 (empty)	±0.25% ±0.25%
Waste Drum Weights	10(Full) 2(empty)	±0.25% ±0.25%
Product Drum Weights	2(full 2(empty)	±0.25% ±0.25
Chemical Purity, Feed (%T)	1(Harshaw lot*)	±0.1%
Chemical Purity, Waste (%T)	1	±0.1%
%T in Spent Carbon and Alumina	See Section VII, Part 14	
Feed Drum X-Assay	1	±1.8%**
Waste Drum X-Assay	6	±1.8%**
Product Drum X-Assay	2	±1.8%**

*One sample is submitted by Harshaw for each lot of feed. This should be analyzed 4 times to secure a precision of 0.05%.

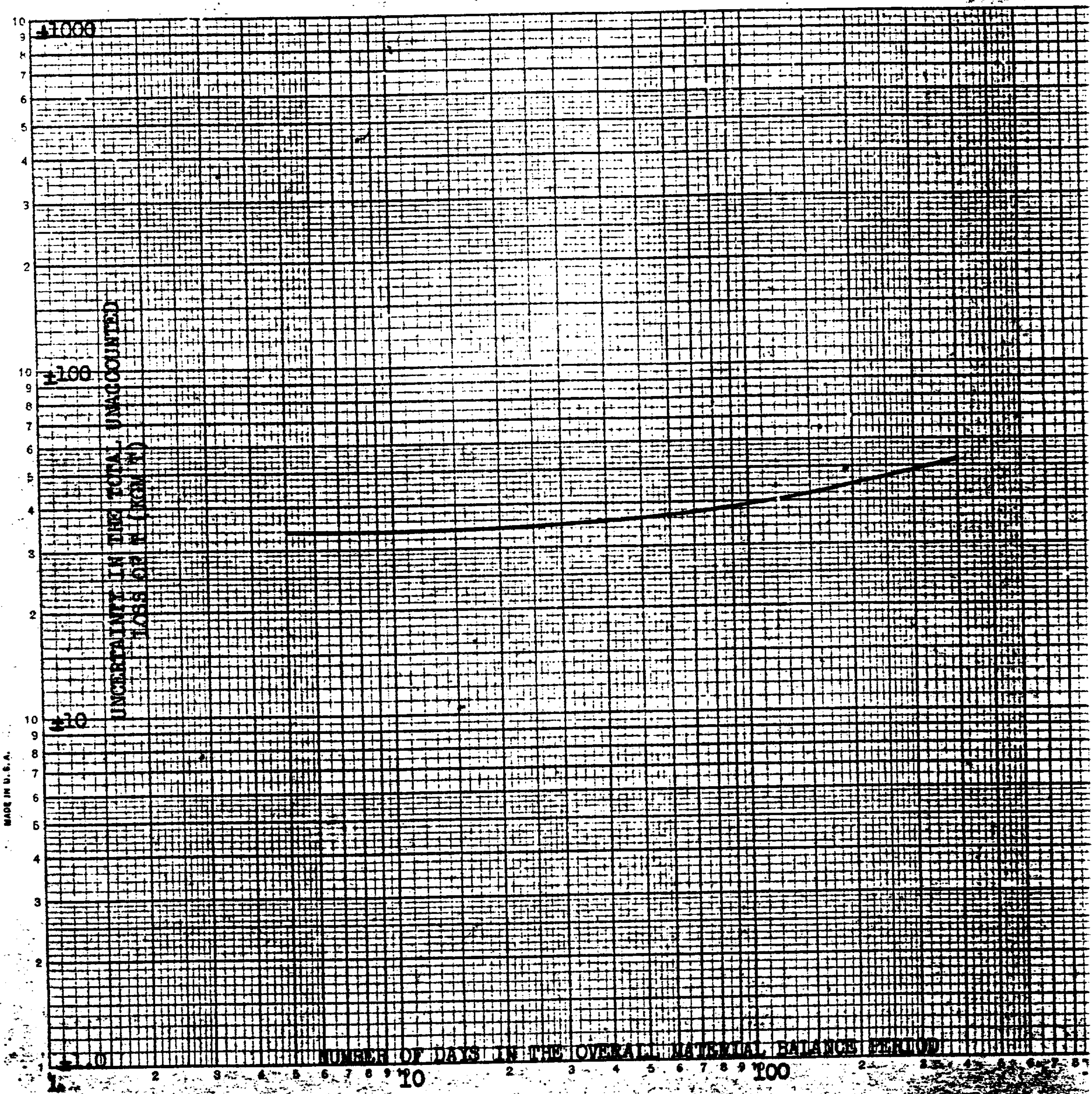
**These are precisions. Various assumptions concerning bias of feed, waste and product assays are listed in Figures 3 and 4.

[redacted]

[redacted]

UNCERTAINTY IN THE TOTAL UNACCOUNTED

LOSS OF T



FIGURE

[redacted]

[redacted]

UNCERTAINTY IN THE DAILY RATE

OF UNACCOUNTED LOSS OF T

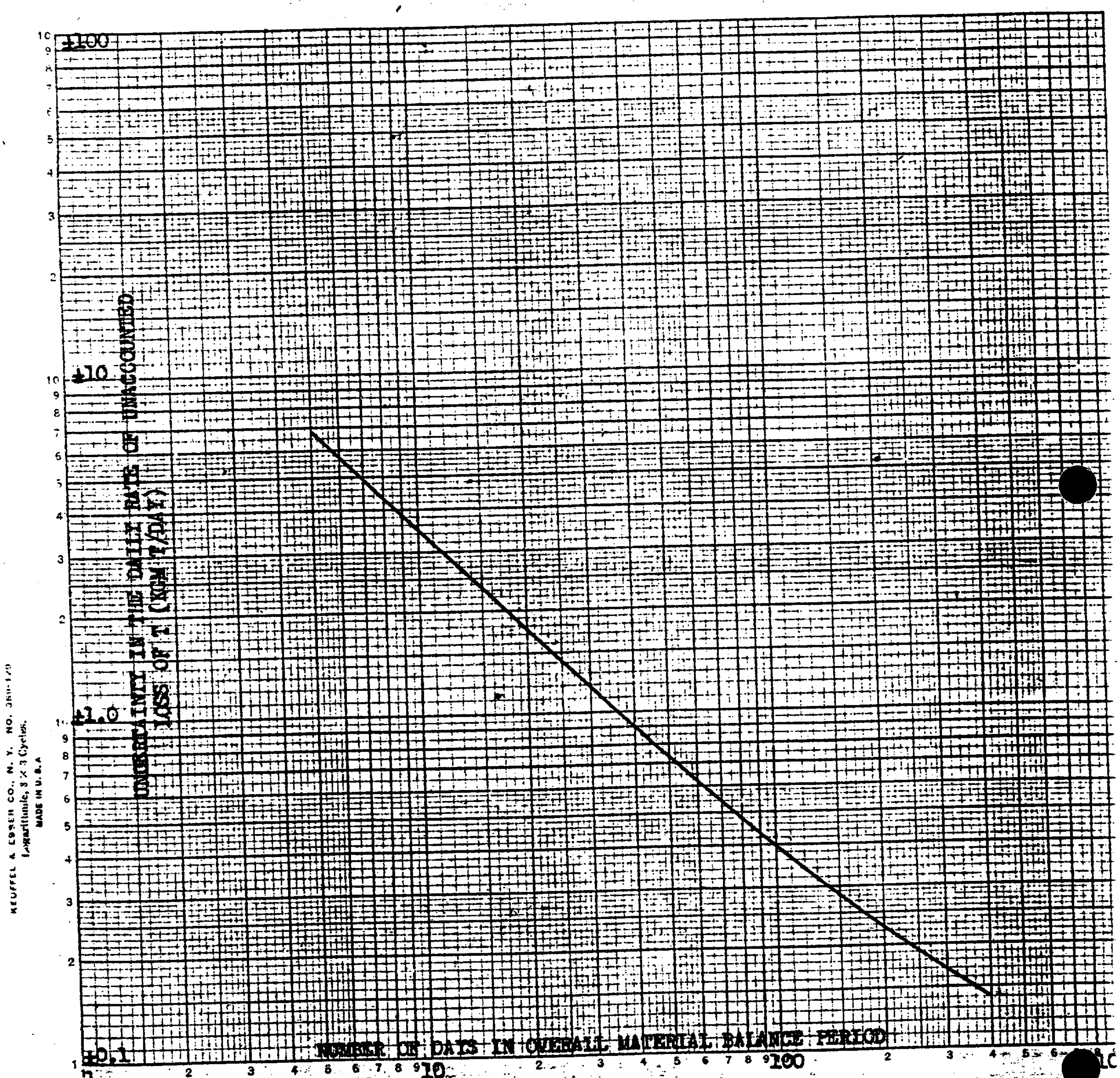


FIGURE 21

[redacted]

[redacted]

UNCERTAINTY IN THE DAILY RATE

OF UNACCOUNTED LOSS OF X

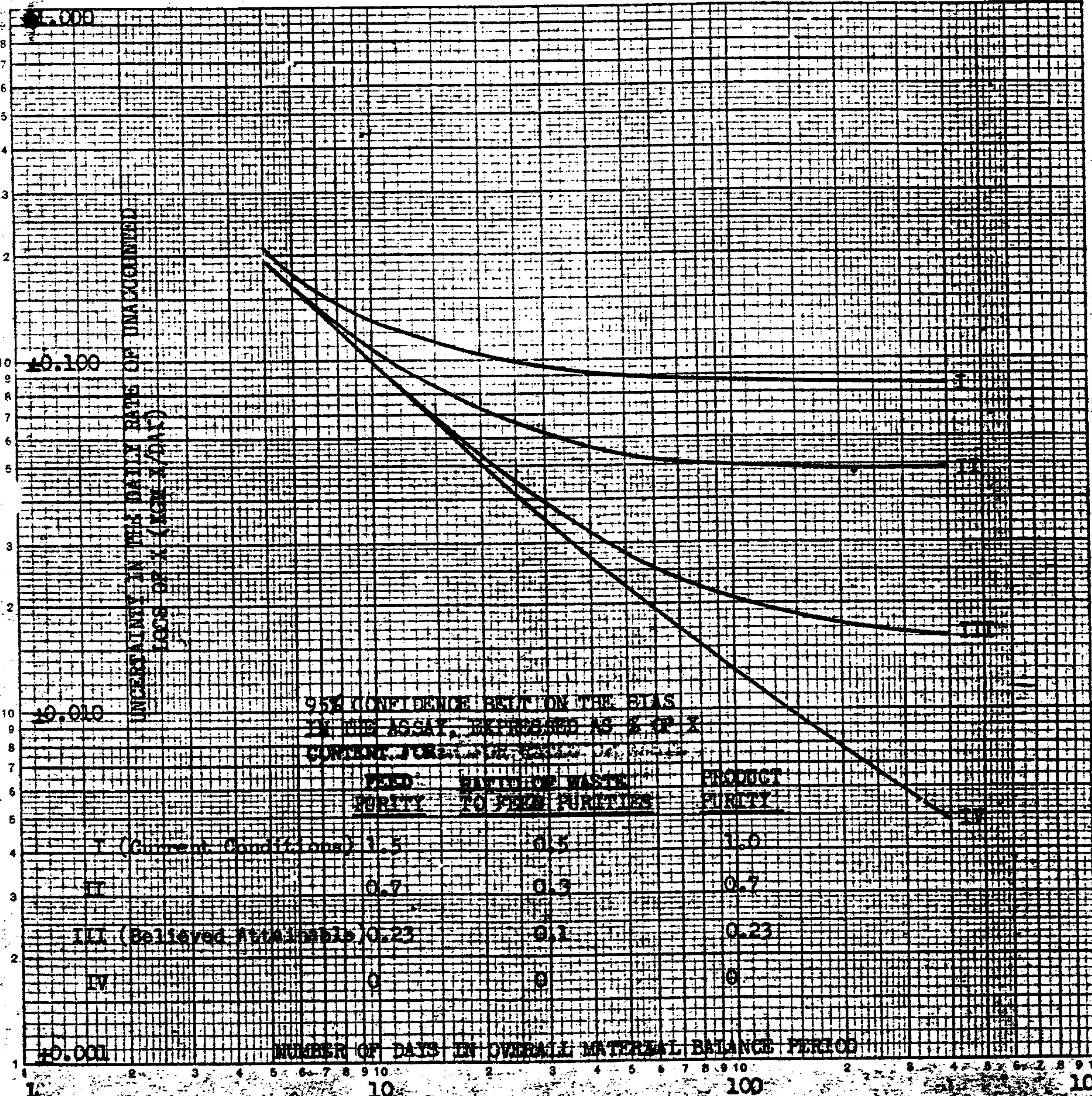


FIGURE 3

[redacted]

UNCERTAINTY IN THE TOTAL UNACCOUNTED

LOSS OF X

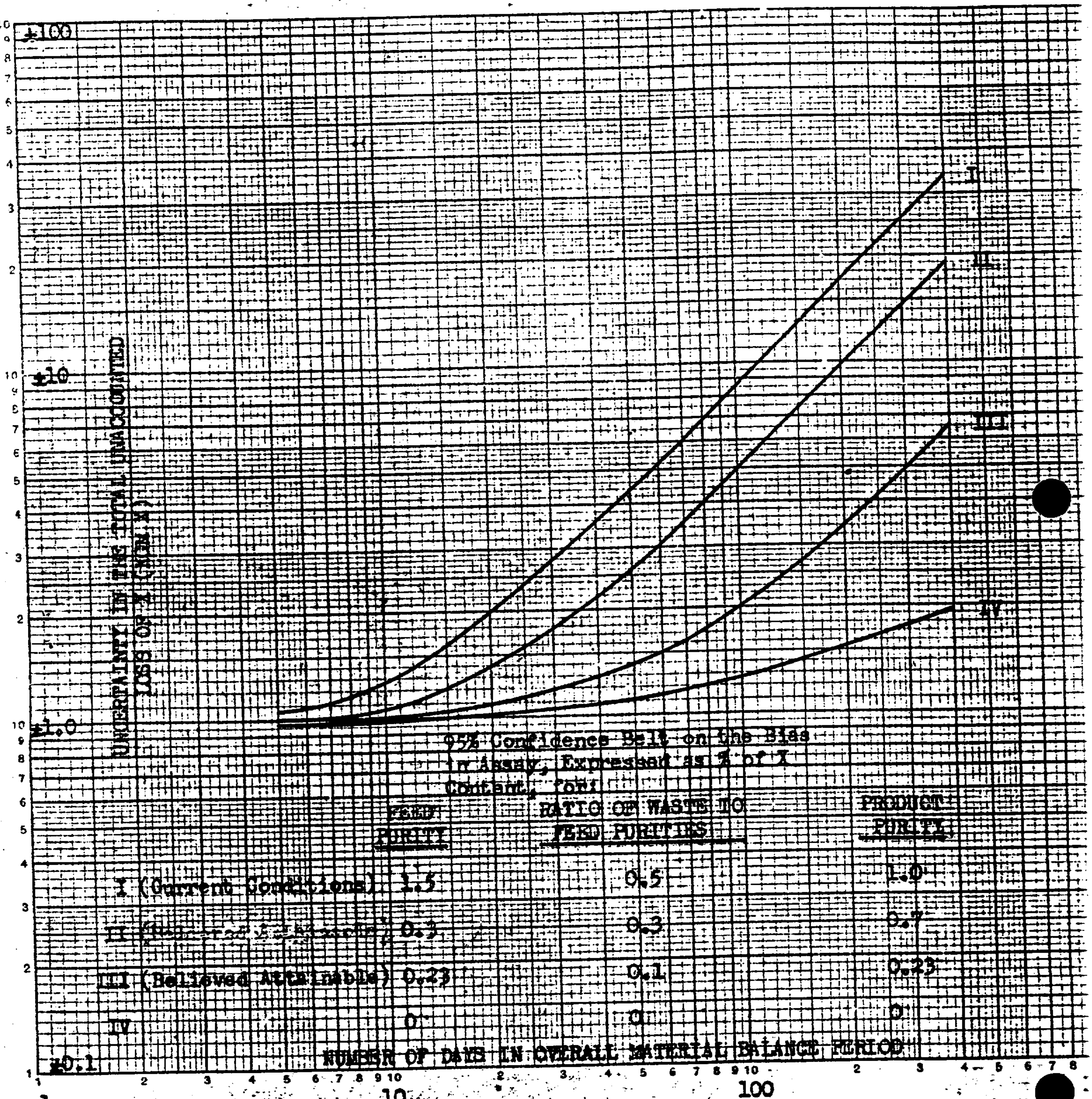


FIGURE 4

[redacted]T

III RECOMMENDATIONS

The principal recommendation of this report is that a reliable material accounting system be set up at the diffusion plant, so that material balances for T and X may be evaluated with the least practicable uncertainty. A program to facilitate establishment of such a system and specific recommendations which it is believed will improve its reliability are described under the headings:

1. Establishment of a Material Accounting System
2. Suggestions for Systematizing Material Accounting.
3. Special Procedures.
4. Additional Technical Studies to Improve Material Accounting System.

The recommendations and program represent the views of the Process Development Department only. They are submitted as a starting point for discussion and revision by the operating departments and the laboratory, who will be primarily responsible for carrying out the material accounting procedures.

1. Establishment of a Material Accounting System

The most important single factor in ensuring the success of a material accounting system is proper organization of personnel and methods for the work. The work should be carried out in accordance with a systematic plan and schedule, with all persons involved following the same set of formal instructions. The following suggestions should be valuable in systematizing material accounting.

[redacted]T

[redacted]

(a) Material Accounting Section - A Material Accounting Section should be organized and charged with the responsibility of setting up the material accounting system, supervising its operation, and reporting its results. The technical staff of this section should be composed of chemical engineers and accountants. It should consult frequently and work closely with the Process Development Department, the Process Division and the Laboratory.

(b) Material Accounting Office - Headquarters of this Section should be located in a material accounting office from which material accounting procedures should be issued, to which the results of material accounting measurements by the operating departments and the laboratory should be forwarded, and from which official reports on plant inventory, yields and material balance should be issued.

(c) Flow Sheet and Terminology - A material flow sheet and terminology for describing material handling should be approved and adopted by the Material Accounting Section, the Process Division, the Laboratory and the Plant Management. Figure 4 of this report is suggested as a point of departure for this purpose.

(d) Detailed Provisional Material Accounting Procedures - The material accounting section should draft detailed provisional procedures for accounting for material in every operation and every transfer represented in the approved Material Flow Sheet. The responsibility for carrying out each procedure should be unambiguously assigned to a specific department, and agreement concerning all details of the procedure should be reached by the department and the Material Accounting Section.

[redacted]

[redacted]

(e) Trial of Procedures - These provisional procedures should be tried for several material balance periods to determine their reliability and to learn where they could be simplified or improved. The results obtained with provisional procedures should be reviewed by the Process Development Department and recommendations made for their revision.

(f) Revision of Procedures - After this trial period, final procedures should be issued and made the basis for the permanent material accounting system.

2. Suggestions for Systematizing Material Accounting

(a) Inventories - The Material Accounting Section should prepare and issue forms showing all information to be recorded in taking inventory of each operation. The usual precautions in taking inventory should be observed to ensure that every container is inventoried once and none twice. The Material Accounting Section should set the exact time at which inventory is to be taken, preferably at four-week or one-month intervals. All vessels whose inventory cannot be determined directly should either be emptied before inventory time or isolated at inventory time to permit discharge and analysis of contents. Vessels should be isolated, inventory measurements observed and inventory samples taken as close to inventory time as possible. At the first inventory it is important that all uninventoriable materials be cleaned out before the first period starts, or carefully segregated from materials subsequently processed.

(b) Transfers - The Material Accounting Section should prepare and issue forms confirming the shipment and receipt of every transfer of material containing T, and listing all information required concerning each transfer. These forms should be filled out and exchanged

[redacted]

[redacted]

by the departments shipping and receiving the material, and copies sent to the Material Accounting Section. All transfers of material or accidental losses should be reported to the Material Accounting Section.

(c) Sampling and Analysis - Whenever samples are sent to the Laboratory for analysis, these should be accompanied by a Request for analysis giving the sample number, a description of the material sampled and the analytical results desired. Reports of analyses from the Laboratory should refer to the sample number and identify the material sampled. Copies of Requests and Reports should be sent the Material Accounting Section. The Section and the Laboratory should develop methods for orderly scheduling of analyses, since a complete material balance cannot be worked up until all analyses have been reported.

Other suggestions for systematizing material accounting are given in Section VIII.

3. Specific Procedures

Tables IX and X of Section VIII summarize specific procedures suggested for determining the T and X associated with each transfer and each inventory given on the Material Flow Sheet. The more important of these procedures are recapitulated below:

(a) Feed - Each drum should be weighed twice before charging and twice after charging on scales with a precision of 1/4%. The sample of each lot of feed submitted by Harshaw should be analyzed for T. A sample of the vapor phase of each drum should be assayed once for X.

[redacted]

[redacted]

(b) Waste - Each drum should be weighed twice before filling and ten times after filling, on scales with a precision of 1/4%. The contents of each drum should be melted and agitated and the liquid phase sampled, analyzed for T and assayed six times for X. Further investigation may make it possible to eliminate melting and agitating drum contents. The present Section 630 waste accumulator sampling apparatus should be given a trial.

(c) Product - For preliminary records the weight of product drums and the assay of the product stream determined at K-25 may be used. Because of the difficulty of securing a representative sample of product drum contents at K-25, for official records, each cylinder or batch of cylinders should be dissolved in a known weight of water at Y-12, and a sample of the solution returned to K-25 for T and X analysis. The number of independent analyses per sample will depend on the number of cylinders dissolved per batch. It would be desirable for a K-25 weigh-master to be responsible for weights of K-25 product obtained at Y-12.

(d) Cascade Inventory - Forms and procedure for cascade T and X inventories are being worked up by the Process Development Department. Two assays should be run on the sample taken from each building for X inventory.

(e) Carbon and Alumina Traps - At each inventory time, all carbon and alumina traps should be dumped except those known to contain less than 0.05 kgm. X.

(f) Decontamination and Recovery Departments - All the T and X in the Decontamination and Recovery Departments at the end of a [redacted] T

material balance period should be converted to T oxides before the departments start processing material from the next period.

When the above procedures call for repeated weighings, these should be statistically independent. That is, each weighing should be made without knowledge of the results of prior weighings.

4. Additional Technical Studies to Improve Material Accounting System -

The material accounting methods suggested in this report have been drawn up without complete data on the precision of all the principal measurements, without complete recommendations on certain procedures and without practical experience with some of the operations recommended. The following technical studies are recommended to supply most of these deficiencies:

(a) Main Process Inventory - The Process Development Department should issue forms for recording all measurements needed in taking inventory of the main and purge cascades and the surge systems of the K-25 and K-27 plants. This department should also issue operational and computational procedures for this inventory. The cell size factors should be determined with a precision of ±3% for sizes 1, 2 and 3 and ±5% for size 4. The precision now and ultimately attainable in the calibration of tails pressure recorders and control valve position indicators should be determined.

(b) Weight of Feed, Product and Waste - The precision and bias of feed, product and waste scales should be checked by cross-weighings during each material balance period.

(c) Product Analysis - The most reliable method of obtaining a representative sample of product and determining its T content appears to be to dissolve the contents of each cylinder or batch of cylinders

[redacted]

[redacted]

in a known wiehgt of water at Y-12, and return samples of the solution to K-25 for analysis and assay. Procedures should be worked out with Y-12.

(d) Feed Assay - Each feed drum should be sampled and assayed for X until the variability of samples and assay has been determined, and the X content of normal feed known to within ±0.1% of the true value. This may permit reduction in the number of drums ultimately to be sampled and assayed.

(e) Waste Sampling - A statistical analysis should be made of the X assay of samples drawn from waste drums agitated for different periods of time, to determine the extent of agitation required, if any.

(f) Sampling Carbon and Alumina - A statistical study should be made of the reliability of procedures for sampling spent carbon and alumina, as discharged from traps, or in drums.

(g) Decontamination and Recovery - A survey should be made of decontamination and recovery operations to determine how to secure the maximum amount of information concerning consumption from these operations.

(h) Consumption - The rate of consumption of T by plant surfaces should be evaluated by examination of laboratory data, by the amount of T recovered in the decontamination of normal equipment and by special analyses of the T present on normal equipment taken out of service specifically for this study.

(i) Combined operations - The analysis of uncertainty in the K-25 main process material balance given in this report should be extended

[redacted]

[redacted]

to the combined K-25 and K-27 plants.

(j) Bias in Assay - The laboratory should seek to reduce the possible bias in X-assay comparing feed, product and waste with synthetic mixtures of known assay made up from stripped U-238 and highly concentrated U-235, from Y-12.

(k) U-234 Inventory - At least one U-234 inventory of the cascade should be made to determine its value as a check on T and X material balances.

[redacted]

IV. FLOW OF T AND X IN THE DIFFUSION PLANT

1. Material Flow Sheet

Flow Pattern - The first step in setting up procedures for accounting for T and X in the diffusion plant is the establishment of a specific pattern for the storage and transfer of T and X in all forms in the K-25 and K-27 plants. The establishment of such a pattern is difficult, and somewhat arbitrary, because material handling procedures have not always been perfectly definite, and because T and X appear in so many different forms· for instance, solid TF_6 in cylinders, gaseous TF_6 in process equipment, solid corrosion products in equipment to be decontaminated, analytical laboratory wastes, and spent carbon and alumina.

The material flow sheet, Figure A, found at the rear of this folder, represents the pattern proposed for describing the storage and transfer of T and X in the K-25 and K-27 plants, for the purpose of the present material accounting study. All subsequent discussion in this report will be keyed to this flow sheet. The flow sheet is also offered to the Engineering, Process, Maintenance and Laboratory Divisions as a basis for systematically routing materials in process and describing the materials handled in the plant.

Operations - The rectangles shown in the flow sheet represent the individual operations into which it is proposed that the diffusion process be broken down for material accounting purposes. Each operation has been given a name and an identifying letter, running from A through Z and supplemented by AA, BB, CC, KA, and KB. With each operation is associated the storage of a definite inventory of T and X, which will vary from time to time, and may

[redacted]

occasionally be zero. These operations must be so defined that all T and X in process in the K-25 and K-27 area is listed, and that no T or X is listed more than once.

Each operation used in this report may be thought of as signifying all material handling or storage steps which may be conveniently grouped into a single concept for material accounting purposes. An operation is not to be thought of as constituting all material handling steps occurring in a particular small area. In some cases one operation will include a number of plant areas; in others, a number of operations will be found in a given area. For instance, all process operations involving the handling of gaseous TF_6 in the main and purge cascades are thought of as a single operation (Item K), even though these are scattered throughout 54 separate process buildings, Section 300; 9 separate buildings, section 400; Section 600 and Section 630. In this instance it is necessary to conceive of all these buildings as a single operation, because of the impossibility of accounting accurately for the flow between buildings. On the other hand, the decontamination of equipment and recovery of T from it in the Conditioning Building is represented by nine operations, because it is convenient to classify these steps in accordance with the type of equipment being handled and the nature of the step, whether decontamination or recovery.

Transfer - The lines connecting the rectangles of the flow sheet represent the routes by which it is proposed to describe the transfer of all T and X from one operation to another, for material accounting purposes. Each line has been given a name describing the material transferred by that route, and an identifying number. With each route is associated the transfer of a definite amount of T and X in a specified period of time. In some cases, this transfer may take place as a steady flow, in others as an occasional shipment of batches of material, and in others no material may be transferred for long periods of time. These routes must be so defined that all material transferred from one operation to another is included, and that no material transferred is counted more than once. In case an operation is by-passed in a transfer of material, as for example in direct shipment of cascade product without intermediate storage (operation E), the transfer is to be assigned to all routes which would have been followed if the operation had not been by-passed, in this case Routes 15 and 5.

Numerical Data - The numerical data given in Figure A for certain operations refer to the approximate inventory of T and X associated with those operations on October 31, 1945. The numerical data given in Figure A for transfer of material by certain routes refer to the approximate amount of T and X transferred from October 1 to October 31, 1945. These data are supplied primarily as examples of the relative importances of the different operations and transfers. Since the data given on the flow sheet pertain to October, 1945, they do not represent the K-27 plant. In particular, the data given for items 18, 19, 28, 29, H and I actually relate to section 600 of the K-25 plant rather than to Section 630 of the K-27 plant. Data have been omitted only where significant measurements of inventory or flow cannot be made, for technical reasons. An example is the inability to measure reliably the amount of T and X transferred from Section 300 to the Conditioning Building with diffusional equipment to be decontaminated (transfer 50). In the procedure for material accounting, no T and X accounts should be kept for the items of Figure A for which numerical data are not given.

The reader may wonder why operations and transfers are represented in Figure A, when no T and X accounting is to be made of them in the material accounting procedure ultimately to be recommended. This has been done for two reasons:

(1) To demonstrate that less error is made by omitting T and X accounts for these operations and transfers than by attempting to include them.

(2) To describe how these operations should be treated at material accounting times to minimize the uncertainty contributed by them to the material balance.

Departments-

The operations represented in Figure A have been grouped into the departments listed below, each enclosed by dashed lines:

(1) Coded Chemicals

(2) Sections 300 and 600

(3) Laboratory

(4) Spent Carbon Storage

(5) Maintenance Services

(6) Recovery

(7) Conversion

This classification of operations into departments is in accordance with the current organization of the Process, Laboratory and Maintenance Divisions of the K-25 plant. In the material accounting procedure recommended in Section VI, it is assumed that each department is responsible for reporting the weight of material, the T content and the X assay stored in each of the operations within the department and transferred by each of the routes whose number falls within the department. For instance, the Coded Chemicals Department would be responsible for reporting the inventory stored in Operations A, B, C, D, E, F and G and the transfers of material by routes 1, 2, 3, 4, 5, 6, 7, 11, 12, 13, 14, 15, 16, 17, 18 and 19. Blocks of adjacent letters and numbers have been assigned to each department, to facilitate location of items.

2. <u>Description of flow, by Departments</u>

a. <u>Coded Chemicals Department</u>

For purposes of T and X material accounting the Coded Chemicals Department is charged with the storage of all relatively pure uranium compounds in the K-25 area. The classification proposed for these compounds and the flow of material of each class are described in the following paragraphs:

(A) <u>Normal TF_6</u>. Normal TF_6 (Code 616) is received (1) in cylinders containing about 300 pounds of technical grade TF_6 of the isotopic content of natural uranium. From storage (A) these cylinders are charged (11) to Section 300. Cylinders containing small residual amounts of TF_6 are shipped (2) from the area for refilling.

[redacted]

<u>(B)</u> <u>Waste TF_6</u>. For present purposes waste is defined as any TF_6 assaying less than 0.65% X. In October, 1945, waste TF_6 entered storage (B), either from the waste accumulator of Section 600 (18) or from the mobile liquefaction unit attached to Building K-311-1 (19). The numerical data given in the figure refer to this type of operation. However, at the present time, waste is normally sent to storage from the Section 630 waste accumulator. The legends in Figure A refer to the present type of operation. At present, no waste is shipped from the K-25 and K-27 area or recycled from storage (B) to Section 300 or 400.

<u>(C)</u> <u>Depleted TF_6</u>. For present purposes, depleted TF_6 is defined as any TF_6 assaying from 0.65 to 0.71% X. Receipts (3) are placed in storage (C) until fed (13) to Section 300. All of the depleted TF_6 now on hand originated as S-50 waste.

<u>(D)</u> <u>Enriched TF_6</u>. Enriched TF_6 is defined as all TF_6 whose assay is between 0.71% X and that of current product. Materials entering storage (D) include enriched TF_6 converted from T oxides at the K-25 area (12) (code 1040), and receipts (4) of S-50 inventory and S-50 product (code 934). In addition storage (D) contains some former K-25 product (codes 1235, 1135, and 935) having a lower assay than current product. Enriched TF_6 cascade feed is charged from storage to Section 300 (14).

[redacted]

[redacted]

(E) Product TF_6 Product TF_6 enters storage (E) as cascade product (15) from section 300 and is then shipped from the K-25 area (5).

(F) Miscellaneous Shipments Occasionally the K-25 plant may be asked to ship small quantities of TF_6 for experimental purposes. The transfer of such miscellaneous materials from Section 300 to Coded Chemicals is denoted by (16), storage in Coded Chemicals by (F) and shipments from the area by (6).

(G) Receipts of Non-Volatile Enriched T The K-25 area has been receiving (7) enriched T from other areas in the following non-volatile forms: Y-12 inventory or product (code 1025) and green salt. This material is charged from storage (G) to the conversion Department (17), where it is converted to TF_6 (code 1040).

In addition to these transfers of T and X shown in Figure A, small amounts of these materials will be shipped from the Coded Chemical department to the analytical laboratory as samples to be analyzed for T and X. These are not shown on the flow sheet but should be included in any material balance.

It is possible to weigh all of the transfers of material to and from the Coded Chemicals Department.

b. Sections 300, 400, 600 and 630.

These sections constitute the main process areas of the K-25 and K-27 plants.

The inventory of T and X in these sections is assigned among the following operations:

[redacted]

[redacted]T

(K) Sections 600 and 630 surge systems and Sections 300 and 400 main and purge cascades

(KA) K-27 Product Storage, to be fed K-25.

(KB) K-25 Waste Storage, to be fed K-27.

(H) Section 630 waste accumulator

(I) Mobile TF_6 Removal unit

(L) Purge and recovery vacuum pumps

(M) Purge and recovery cold traps

(N) Carbon traps of Sections 300, 400, 600 and 630.

(J) Alumina traps of Section 300.

The flow of material to and from these operations will be described in turn.

(K) <u>Sections 300 and 400 Main and Purge Cascades and Sections 600 and 630 Surge Systems.</u>

Operation (K) consists of all parts of Sections 300, 400, 600 and 630 in which gaseous TF_6 is treated, with the exception of the cold trap purge and recovery systems. The inventory of this part of the plant has been termed "Active Inventory" by the Operating Control Department. All these geographically separated parts of the plant have been grouped together for material accounting purposes because of the impossibility of measuring the rate of flow of gaseous TF_6 from one of these parts to another with sufficient precision for material accounting purposes. The inventory of this part of the plant can be reliably determined by volume calibrations and measurements of TF_6 pressures, temperatures, G-74 content, and assays.

[redacted]T

[redacted]

TF_6 is transferred to or from Operation (K) in drums or containers which can be accurately weighed and sampled by the following routes:

<u>From Coded Chemicals</u>

Normal TF_6 Cascade Feed (11)
Depleted TF_6 Cascade Feed (13)
Enriched TF_6 Cascade Feed (14)

<u>To Coded Chemicals</u>

Cascade Product (15)
Miscellaneous Cascade Withdrawals (16)

<u>To Analytical Laboratory (0)</u>

Laboratory Samples (20)

<u>Within Sections 300, 400, 600 and 630</u>

K-27 Product (20A), to or from storage (KA)
K-25 Waste (20B), to or from storage (KB)

In addition to these transfers, which can be accurately measured, sampled and assayed, the following transfers to and from Operation (K), the main and purge cascades and the surge systems, occur in such a form that the amounts of T and X transferred cannot be readily determined.

<u>Within Sections 300, 400, 600 and 630</u>

Condensed Section 630 Waste (28)
TF_6 to Mobile Unit (29)
TF_6 fed to purge and recovery vacuum pumps (21)
TF_6 returned from purge and recovery cold traps (23)

<u>To or from Other Departments</u>

TF_6 discharged with conditioning gases (41)
T shipped in Converters to be unplugged or decontaminated (40)
T returned with fluorinated converters (43)
T shipped in contaminated diffusional equipment (50)
T returned with decontaminated diffusional equipment (53)
T present in contaminated miscellaneous materials (70)

[redacted]

[redacted]

Materials thus transferred must be processed further before the T and X in them can be reliably accounted for. Subsequent paragraphs describe the further processing of these materials.

(H) Section 630 Waste Accumulator Condensed Section 630 Waste (22) runs into the Section 630 Waste Accumulators (H). Liquid waste (18) is drained from the waste accumulators into waste cylinders, each holding about 3000 pounds of TF_6. No accurate measurement can be made of the amount of TF_6 drained from the waste condenser, and no representative assay can be obtained of this stream because of its varying composition and indeterminate flow rate. Also, no accurate measurement can be made of the inventory of the waste accumulator, because the level indication is not sufficiently precise; furthermore it has not been established that a representative sample can be taken of its contents, because of the varying waste composition and the absence of means for agitating its contents.

To obtain a reliable measure of the amount of T and X withdrawn through the waste accumulators (H) it will thus be necessary to drain the accumulators completely at each material accounting period, and to weigh, sample and analyze the contents of each drum of waste shipped (18) to storage.

(I) Mobile TF_6 Removal Unit In case the waste condenser (23) is not in operation, the mobile TF_6 removal unit (I) is used to withdraw gaseous TF_6 of waste composition from the bottom onstream cells of building K-402-1 (29). This TF_6 is compressed and liquefied, stored temporarily in a dispensing drum of the mobile unit and ultimately drained (19) into waste cylinders in the same manner as waste (18) from the Section 630 waste accumulator. Here, also, no accurate measurement can be made of the amount of TF_6 charged to the mobile unit and no representative assay can be taken of this stream because of its varying composition. No accurate measurement can be made of the TF_6 inventory of the mobile unit because the level indication is not sufficiently precise, and no representative sample can be taken of its contents because of the varying waste composition and the absence of means for agitating its contents.

To obtain a reliable measure of the amount of T and X withdrawn through the mobile unit (I), it will thus be necessary to drain the unit completely at each material accounting period, and to weigh, sample, and analyze the contents of each drum of waste shipped (19) to storage.

(L) Purge and Recovery Vacuum Pumps

Gases evacuated (21) by purge and recovery system vacuum pumps (L) contain variable amounts of TF_6. The amount of T and X transferred from the main and purge cascade and surge systems in this way cannot be readily determined, because of the variable TF_6 content and flow rate of these gases.

There are two types of T and X inventory present in purge and recovery vacuum pumps:

(1) TF_6 dissolved as such in pump oil.
(2) Solid T compounds present as dust and sludge in pumps, mist filters and pump oil.

The inventory of dissolved TF_6 can be reduced to a low value by purging the pumps with nitrogen at material accounting times and can be determined by analyzing filtered samples of pump oil. A numerical estimate of the inventory of dissolved TF_6 is given for Operation (L) in Figure A.

[redacted]

The T and X inventory present as sludge cannot be reduced by purging the pumps and cannot be accurately determined except by opening and decontaminating the pumps. This material constitutes one of the hidden losses of T, similar to the deposition of lower T fluorides in the main cascade. Like them it is one of the items constituting losses of T and X to be evaluated by material balance on the process area.

T and X may leave the purge and recovery vacuum pumps in one of two ways:

As TF_6 fed to refrigerated cold traps (22).
As sludge or dissolved C-616 in contaminated vacuum pumps and oil (60) shipped to Maintenance Service for decontamination.

T may be returned to purge and recovery vacuum pumps in decontaminated vacuum pumps and oil (63). None of these transfers of T and X is susceptible to even approximate measurement. No reliable measurement of TF_6 fed to refrigerated cold traps (22) is possible because of the widely varying rate of flow and TF_6 content of these gases.

(M) Purge and Recovery Cold Traps

The inventory of purge and recovery cold traps (M), consists of the solid TF_6 condensed in them and reduced T fluorides. Only the former material can be returned to the cascade (23) by vaporization. The latter consists of hidden inventory like the lower T fluorides deposited in Sections 300, 400, 600 and 630 and in vacuum pump sludge. A small amount of TF_6 is lost from refrigerated cold traps (24) and (25), remaining uncondensed at the temperature of the gases leaving the cold traps. Gases

[redacted]

leaving cold traps flow either through carbon traps (24) or alumina traps (25). The amount of T in these gases can be estimated from the total valume of nitrogen flowing through the cold traps and the refrigerant temperature.

(N) Carbon Traps

Carbon Traps (N) are used to absorb TF_6 from gases (24) vented from cold traps in all sections of the plant except at purge points and in Sections 304, 305, 306 and 312. In addition, TF_6 from a variety of types of process equipment may be vented directly to carbon traps when relief valves open or equipment is purged. No reliable estimate of the amount of TF_6 thus transferred to carbon can be made, because of the intermittent, variable character of the flow.

It may be assumed that gases vented from carbon traps (27) contain substantially no TF_6, provided the carbon trap charges are renewed when trace indicators show that absorption is starting to be incomplete.

An approximate measure of the T inventory of some carbon traps (N) may be secured from the increase in weight of these traps during service. In other carbon traps, an approximate measure of the T inventory can be secured by means of gamma ray absorption measurements. Neither of these methods of determining T inventory is regarded as reliable, since both are affected by other materials which may be picked up by the traps, such as vacuum pump oil, and the gamma ray method may be in error when the distribution of T throughout the trap is not uniform.

The amount of T and X present in spent carbon (30) discharged from carbon traps can be determined by adequate weighing, sampling and analysis of such materials.

[redacted]

(J) Alumina Traps

At present, gases (25) vented from cold traps at purge points and in Sections 304, 305, 306 and 312 are passed through alumina traps, rather than carbon traps, to absorb traces of TF_6. Carbon traps are not being used at these points because C-215 potentially present in these gases might react with carbon to form an explosive compound. A crude estimate of the amount of T and X thus transferred to alumina traps can be secured from the volume of gases vented and the cold trap refrigerant temperature.

The amount of T and X vented from alumina traps (26) cannot be reliably estimated at present, because recovery of TF_6 by alumina is not so complete as by carbon, and the factors controlling the degree of recovery have not been fully evaluated. Gases vented from alumina traps (26) thus constitute one source of loss of T and X, which, although small, cannot now be evaluated. The loss shown at this point in Figure 1 was estimated by assuming 70% recovery of TF_6 by alumina traps.

An approximate measure of the T inventory of alumina traps (J) may be secured from the increase in weight of these traps or by gamma ray absorption measurements. Neither of these methods of determining T inventory of alumina traps is regarded as reliable, for same reasons as with carbon traps.

The amount of T and X present in spent alumina (34) discharged from alumina traps, can be determined by adequate weighing, sampling and analysis of materials.

[redacted]

[redacted]T

c. C-216 Disposal Plant

In the past, from time to time, an indeterminate amount of TF_6 has been discharged with conditioning gases (41) from Section 300 and pumped to the C-216 disposal tower, K-1405 (3). This practice has been discontinued, possibly permanently, but is here noted because it constitutes one possible indeterminate source of loss. ith the solution of NaOH now used in this tower, most of the T and X pumped to it is precipitated and settles out at various locations, thus making accurate sampling and material accounting impossible. If TF_6 is to be pumped to K-1405 in the future, a solution of Na_2CO_3 should be used in place of NaOH, so that the T will remain in solution and be more readily accountable.

d. Maintenance Services

For material accounting purposes, it is proposed that equipment received by maintenance services for removal of T (decontamination) be classified as:

(50) Contaminated Diffusional Dequipment, consisting of all equipment normally used in the main and purge cascades and the surge systems (Operation K). This will include all pumps except Beach Russ pumps, piping, valves, gas coolers, drums, etc., and all diffusers except those to be reclaimed by fluorination. Fluorinated converters which must be further decontaminated (45) are also handled by Maintenance Services.

(60) Contaminated Vacuum Pumps and Oil, consists of Beach Russ pumps for C-616, and oil and sludge from such pumps, from Sections 300 and 400 purge and recovery systems, Section 630, Section 413, and mobile TF_6 removal units.

(70) Contaminated Miscellaneous Materials from Sections 300, 400, 600 and 630, consisting of spillage, process area gatherings, floor sweepings, dust from vent lines and all other sources of T than those specifically enumerated.

[redacted]T

(80) Contaminated laboratory Wastes and Equipment, consisting of scrapped laboratory equipment, waste solutions, and all other T containing materials discarded by the laboratory.

Each of these types of material is cleaned of T (Decontaminated) in operations (R), (T), (V) and (X), and the decontamination solutions and other materials in which T is concentrated are transferred to the Recovery Department for extraction of T and X.

Whatever the present practice of the Maintenance and Recovery Departments, it is desirable that each of the four types of contaminated material ((50), (60), (70) and (80)) be processed separately through the points where the recovered T oxides are weighed and sampled ((52), (62), (72) and (82)). This is recommended so that the T and X recovered may be properly credited to the four types of material entering Maintenance Services for decontamination.

Maintenance Services will return some decontaminated diffusional equipment (53) containing an indeterminate amount of T and X to Sections 300, 400, 600 or 630 and will occasionally scrap some diffusional equipment (54). In the case of equipment to be scrapped, it is important for material accounting purposes that all detectable traces of T be removed from such equipment. Similarly some decontaminated vacuum pumps and oil (63) will be returned to purge and recovery systems, and some will be scrapped (64). Complete recovery of T from such scrapped pumps is also imperative.

e. Recovery Department

The Recovery Department receives from the Maintenance Services Department decontamination solutions from four types of material.

(51) From diffusional equipment.

(61) From vacuum pumps and oil.

(71) From miscellaneous materials.

(81) From laboratory wastes and equipment.

These decontamination solutions consist of washings, scrapings dust, sediment, and all other forms in which T and X are removed from equipment decontaminated by Maintenance Services. Because of the heterogenous and miscellaneous character of these solutions, it is probable that they cannot be reliably sampled at these points. In the Recovery Department, each of these types of decontamination solution should be processed separately, and the T and X concentrated and recovered in a form which can be readily weighed, sampled and analyzed, such as T oxides. After sampling, these T oxides are combined and shipped to the Conversion Department for conversion to TF_6. The four types of T oxides prepared by the Recovery Department are:

(52) T oxides from diffusional equipment.

(62) T oxides from vacuum pumps and oil.

(72) T oxides from miscellaneous materials.

(82) T oxides from laboratory wastes.

Material in each of these four recovery lines should be kept separate until the T oxides have been weighed and sampled, so that the proper amount of T may be credited to each of the sources from which the Maintenance and Recovery Departments receive T.

The Recovery Department also recovers T by direct fluorination of converters. Contaminated converters to be fluorinated (40) are received from Sections 300 or 400 (K) and fluorinated (Q) to unplug the barrier tubes and render the converter suitable for reuse in the cascade. TF_6 produced in this operation (42) is weighed, sampled, assayed combined with other TF_6 converted in the K-25 Area (12), and sent to the Coded Chemicals Department. Fluorinated converters (43), from the fluorination operation are returned to Sections 300 and 400 if they are suitable for reuse. If they are not suitable for reuse and contain no more T, they are scrapped (42). If they are not suitable for reuse, but still contain T, they must be decontaminated like other diffusional equipment, in operation (E). In this case, the converters may be considered shipped to Maintenance Services by route (45).

f. Laboratory

Inasmuch as around 200 grams of X per month are shipped to the Laboratory (C) in laboratory samples (20), it is desirable that an approximate record be kept of the weight of every sample received by the laboratory. When these samples have been analyzed and assayed, it will be possible to compute the amount of T and X present in all material shipped to the laboratory.

The flow sheet indicates that the laboratory received samples only from Sections 300, 400, 600 and 630 (Operation (K)); most of the X entering the laboratory comes from this source. However, small amounts of X will also be received in samples from many of the other numbered and lettered points in the Figure.

Outgoing material from the laboratory consists primarily of waste solutions and scrapped equipment. In the flowsheet, it has been assumed that these will be decontaminated by Maintenance Services and the T recovered by the Recovery Department. It is possible that much of the decontamination will be done in the laboratory and that some solutions will be shipped directly to the Recovery Department, operation (Y).

g. Spent Carbon and Alumina Storage

Like the Coded Chemicals Department, spent Carbon and Alumina Storage acts only as a shipping, receiving and storage department, and does not process T and X. Its only receipts are spent alumina (34) from Section 300 and spent carbon (30) from Sections 300, 400, 600 and 630. Receipts of this character should be weighed and sampled by procedures proved reliable.

From Storage (P) spent Carbon and Alumina may be shipped by one of three routes:

(1) At present, all spent carbon containing less than the natural assay (0.71%) of X is shipped (32) out of the accounting area to dead storage.

(2) Some of the spent carbon and alumina containing more than the natural assay of X is shipped off the K-25 area (33) for recovery of T and X.

(3) The balance of the spent carbon and alumina containing more than the natural assay of X is shipped (31) to the Conversion Department for Recovery of T and Conversion to TF_6.

h. Conversion Department

The Conversion Department receives three types of material for conversion to TF_6:

T oxides from the Recovery Department (52), (62), (72) and (82).

Spent carbon and alumina (31) from Spent Carbon and Alumina Storage, and

Non-volatile T (17) from the Coded Chemicals Department.

[redacted]

Because the conversion yield from each of these types of material may be different, it is suggested that each be processed separately by the Conversion Department, as follows:

Recovered T oxides, operation (AA).

Spent carbon and alumina, operation (BB).

Non-volatile T from Coded Chemicals, operation (CC).

TF_6 prepared by these three operations is described as:

TF_6 from Recovered T Oxides (91)

TF_6 from Spent Carbon and Alumina (92)

TF_6 from Non Volatile T Receipts (93).

These separate sources of TF_6 are combined as TF_6 converted at K-25 Area (12) and shipped to the Coded Chemicals Department.

[redacted]

[redacted]

V. GROUPING OF OPERATIONS FOR MATERIAL BALANCE

1. Introduction

The grouping of operations for material balance purposes must take into account the two main purposes for establishment of the material accounting system: To provide an account of all material committed to the K-25 Area, so that illegal diversion of X in amounts exceeding a specified quantity will not go undetected; and to estimate the losses of T and X within the main process, so that plant performance and the corrosion properties of materials of construction can be better evaluated. Other purposes are the evaluation and localization of losses within specific departments.

Of the many individual operations or groups of operations shown in Figure A for which material balances may in theory be evaluated, only a few can be used in practice. This is primarily a consequence of the fact that reliable material balances cannot be evaluated when there are one or more routes to or from a group of operations for which the amount of material transferred cannot be accurately determined.

The purpose of this Section is to suggest a grouping of operations for which reliable material balances may be evaluated, which it is thought will provide adequate knowledge of the nature and location of losses without requiring an excessive number of individual balances. It is suggested that the following material balances be computed:

[redacted]T

[redacted]

Overall Material Balance

Main Process Material Balance

Laboratory Material Balance

Conversion Department Material Balance

Coded Chemicals Material Balance

Spent Carbon and Alumina Material Balance

2. Overall Material Balance

The overall material balance on the entire group of operations of the K-25 and K-27 plants evaluates all losses from these plants, and is thus important as a general check on operations. Since no information is provided concerning the distribution of these losses among the various operations and departments, such a material balance obviously must be supplemented by more detailed balances on less extensive portions of the plant.

The following quantities enter the overall material balance:

The inventory of all plant operations at the beginning and end of the material balance period

The amount of material transferred during the material balance period by the following routes:

(a) Transfers which can be accurately weighed, sampled and analyzed

(1) Receipts of normal TF_6
(2) Feed cylinder residues
(3) Receipts of depleted TF_6
(4) Receipts of enriched TF_6
(5) Product shipments

~~SECRET~~

[redacted]

(6) Miscellaneous shipments

(7) Non-volatile T receipts

(32) and (33) Spent carbon

(b) Transfers assumed to contain no T

(27) TF_6 from carbon traps

(44), (54) and (64) Scrapped equipment

(c) Transfers of small amounts of T which cannot be accurately determined

(26) TF_6 from alumina traps

3. Main Process Material Balance

The portion of the plant for which a material balance will give the most valuable process information consists of those portions of Sections 300, 400, 600 and 630 handling gaseous TF_6. It is probable that the principal unaccounted losses of T and X will be found in this part of the plant, because of "consumption", the reduction of TF_6 to non-volatile T fluorides by reaction of TF_6 with plant surfaces. Losses from other causes will also occur in this part of the plant, but reduction of TF_6 will be the principal one. A material balance on this part of the plant will, therefore, set an upper limit to the rate of reduction of TF_6 by metal surfaces, provided the corrections described in Section VI are applied.

To minimize other causes of loss than reduction of TF_6, it would be desirable to include only the main and purge cascades and the surge systems of K-25 and K-27 plants (Operation K) in the Main Process Material Balance. Unfortunately, a larger number of operations must be used, because many of the transfers to or from Operation K cannot be accurately measured. Such transfers include:

[redacted]

[redacted]

(21) TF_6 fed to Purge and Recovery Vacuum Pumps

(23) TF_6 returned to Cascade from Cold Traps

(28) Condensed Section 630 Waste

(29) TF_6 to Mobile Unit

(40) TF_6 in Contaminated Converters, etc.

From the discussion of the preceding Section it has been concluded that it is necessary to group all the operations within the heavy solid line of Figure A to permit evaluation of a reliable material balance on the Main Process. This is unfortunate, because the unaccounted losses resulting from such a material balance will also include reduction of TF_6 in vacuum pumps and cold traps, losses in decontamination and recovery operations and losses from carbon and alumina traps, besides the reduction of TF_6 in process equipment, the quantity of greatest interest. However, if the boundary of the region over which the Main Process Material Balance is taken were to be contracted from that given in the Figure, it would cut one or more transfers which could not be reliably evaluated, so that there would be a large error in the unaccounted loss evaluated by the material balance. In effect, it is considered preferable that this unaccounted loss be known as reliably as possible, even though it includes several causes of loss besides the one of greatest interest, rather than that it be inaccurately known and include fewer causes of loss.

4. Laboratory Material Balance

The primary object of the laboratory material balance is to determine how much of the T and X shipped to the laboratory for T analysis and X assay is recovered and returned to the process. This is done by grouping the following operations:

[redacted]

(O) Laboratory

(X) Decontamination of Laboratory Wastes and Equipment

(Y) Recovery of T from Laboratory Wastes and Equipment

and determining the transfers to the laboratory by route (20) and the recovery of T oxides from laboratory wastes by route (52).

5. Conversion Department Material Balance

Operations (AA), (BB) and (CC) are grouped for the Conversion Department Material Balance.

The primary object of the Conversion Department Material Balance is to determine how much of the non-volatile T and X concentrate shipped to this department is converted to TF_6 which may be returned to the main process. Material balances on these operations may be for each operation (AA), (BB), or (CC) individually, or for all three of them together. The latter will probably be the more useful balance; the region over which the balance is taken is then identical with that shown for the Conversion Department in Figure A.

6. Coded Chemicals Material Balance:

The primary function of the Coded Chemicals Department is the storage of relatively pure T compounds. This department does not process TF_5 or convert one T compound into another. It may, however, transfer material from one container to another, or reweigh or reanalyze containers, so that a material balance on its operations is also important.

Operations grouped for this material balance include all shown in Figure A within the Coded Chemicals Department and two storage operations of Sections 300 and

(KA) K-27 Product Storage to Be Fed K-25

(KB) K-25 Waste Storage to Be Fed K-27

7. <u>Spent Carbon and Alumina Material Balance</u>

Like the Coded Chemicals Department, the Spent Carbon and Alumina Storage Department is primarily concerned with storage of these particular materials. However, a material balance on its operations is also desirable, because of possible losses during handling and bookkeeping losses arising from resampling or reanalyzing materials.

The material balance will be taken over the region assigned to the Spent Carbon and Alumina Storage Department, in Figure A.

VI. SIGNIFICANCE OF MAIN PROCESS MATERIAL BALANCE: CONSUMPTION AND DIVERSION

The two most important possible causes of disappearance of T and X within the main process material balance envelope are the "consumption" of TF_6 by plant surfaces and the unreported diversion of T and X from this part of the plant. Each of these causes will have an effect on the unaccounted loss of X evaluated from the main process material balance, but neither can be directly evaluated from the loss without other information or assumptions. The purpose of this Section is to discuss consumption and diversion, their interrelation, and methods for evaluating them from the main process material balance.

When the main process material balance envelope is drawn as indicated in Figure A, the unaccounted loss of T or X is made up of the terms given in the following equation:

Unaccounted loss = Unreported diversion
+ Consumption in Section 300, 400, 600 and 630
+ Consumption in Vacuum pumps and Cold Traps
+ Losses in Vent Gases (26) and (27)
+ Losses in Decontamination or Recovery Departments
+ Unrecovered spills or other accidents
− TF_6 recovered from converters (42)
− TF_6 recovered from diffusional equipment (52) and vacuum pumps and oil (62)

In this equation it is assumed that all measurable changes in inventory have been included in the material balance. For example, it is assumed that changes in inventory in the Mobile TF_6 Removal Unit (I), the waste accumulator (H) and Cold Trap (M) are properly evaluated by the procedures outlined in Section VIII, that vacuum pumps (L) are purged and carbon traps (N) and alumina traps (J) are dumped at the beginning and end of the period, and that the inventory of the decontamination and recovery departments is

[redacted]

evaluated as described in Section VIII.

The first three terms of this equation are of primary interest in the present discussion. Estimation or evaluation of the remaining terms is evidently a necessary step to determining the sum of the first three or their individual values.

Losses in vent gases should be kept negligibly small, if possible, by the use of adequately sized carbon or alumina traps and trace indicators to show when these traps are beginning to pass significant amounts of T or X. If it is impossible to keep these losses negligibly small, the total amount of gas purged through each trap should be measured by a gas meter and a rough estimate should be made of the T and X content of the vent gases by trace indicators, space recorders or other suitable analytical devices.

Processes in the decontamination and recovery departments should give substantially complete accountability of T and X. It is desirable but not essential, that the useful recovery of T and X as T oxide be 100%. In any event it should be possible to show where all the T and X goes. Occasional calibration of these processes by putting through known amounts of T is desirable. Demonstrated losses of T and X may then be used to evaluate the fifth term of the foregoing equation.

If unrecovered spills or other accidental losses of

[redacted]

T and X occurs, they should be reported to the material accounting office and the best possible estimate of the amount lost should be made and used for the sixth term of the equation.

TF_6 recovered from converters, diffusional equipment and vacuum pumps can be determined from transfers (42), (52) and (62) from the recovery department. These transfers will be largest when a large amount of equipment which has been on stream for a long time is shipped from the cascade for decontamination and when a high degree of recovery of T is secured in the decontamination operation. If consumption and possible diversion proceed at a steady rate, the unaccounted losses will be lowest, or even negative, when the decontamination and recovery departments are most active and efficient.

If losses in vent gases, the decontamination and recovery department and unrecovered spills are negligible, or have been allowed for, the foregoing equation may be rewritten:

Possible unreported diversions

+ Consumption in Sections 300, 400, 600 and 630

+ Consumption in Vacuum Pumps and Cold Traps =

Unexplained losses of T(orX) = Unaccounted losses of T(orX) + TF_6 reported in transfers (42), (52) and (62).

Thus, a definite procedure is available for evaluating the sum of possible diversion and consumption, which will hereafter be called "unexplained losses". To distinguish between them, further information is necessary.

[redacted]

Normal consumption proceeds at a steady rate. However, if a leak of wet air occurs or if external mechanical friction develops, as with a rubbed impeller, the consumption rate locally may be greatly increased. Reports of such occurrences should be sent the material accounting office, so that a temporary increase in consumption may be expected and explained. Except where such abnormal consumption occurs, "unexplained losses" should be relatively steady, if there are no diversions. If these losses vary excessively, by an amount greater than can be explained by the imprecision of the material balance and possible unreported losses, the existence of a diversion varying in amount from one material balance period to the next may be suspected. On the other hand, a steady value for the "unexplained losses" is not sufficient evidence of non-diversion, because a clever person would try to take the same amount each period and thus escape detection in this way. Thus, all that can be said with certainty is that if the "unexplained losses" are steady, they set an upper limit for the rate of diversion plus the rate of consumption. Additional information is needed to distinguish between the two.

This may be supplied in part by attempting more direct measurements of consumption and in part by the apparent concentration of X in the "unexplained losses".

Three methods of measuring consumption more directly than by material balance are available; all should be used. One is the measurement of consumption of IF_6 by small samples of plant materials in the laboratory. A large mass of data of this character is available, but it is difficult to interpret because the materials are not identical with plant materials and the conditions of exposure, particularly the flow of gases and the concentration of contaminants, differ between plant and laboratory. The second method is by inference from the amount of T recovered in the decontamination and recovery departments from the usual run of equipment down for repair. For this purpose, records should be kept of the nature of all equipment decontaminated in each batch, the length of time and the part of the plant in which it was exposed to TF_6, and any unusual circumstances connected with its service, which might make it not representative of the rate of consumption. This method is apt to be misleading, because equipment removed from the cascade for repair may well have experienced an abnormally high consumption rate and each type of equipment will not be removed in proportion to the number installed in the cascade. The greater the number of classes into which equipment to be decontaminated is segregated, and the more detailed the records kept by the decontamination and recovery departments, the more useful will be the information secured by this method. The classification of material and equipment to be decontaminated into the four groups noted in Figure A is only the minimum useful degree of classification.

The third method is by removing all the equipment of several normal cells which have been operated under known conditions and by making a careful analysis of the T deposited in this equipment.

[redacted]

After data of these three types have been collected and studied, a fairly reliable estimate of the losses of T and X can be made. If the difference between the estimates and the "unexplained losses" exceeds the precision of these quantities with statistical significance, unreported diversion may be suspected.

The apparent concentration of X in the "unexplained losses" (i.e., the ratio of the X losses to the T losses) can be estimated from laboratory consumption data, recovery data, and estimates of plant surfaces with greater precision than the actual magnitude of the losses themselves. If the ratio obtained by material balance differs with statistical significance from the predicted ratio, diversion will be suspected. It will be noted that a diverter cannot restore X of low purity to the plant in order to cover up diversion of high purity without greatly increasing the ratio of X loss to T loss.

It should be noted that the diversion of material from the recovery and decontamination operations is particularly hard to detect. The material entering these operations during a given material balance period has largely already been reported as an "unexplained loss" in earlier material balance periods; this material has already dropped from the sight of the material accounting system. It is recovered, and reported in transfers (42), (52) and (62). The unexplained loss for the material balance period in question is obtained by adding these transfers to the unaccounted loss for the period. If unreported material is diverted from the recovery operations during the period, the

[redacted]

amount of material in transfer (42), (52) and (62) will be smaller, and the unexplained loss smaller. If at the same time an equivalent amount of X is diverted from Section 300, the unaccounted loss of X during the period will be larger, and the total unexplained loss of X may well be the same as in earlier material balance periods during which no unreported diversion took place. In this way it might be possible for material to be diverted for a large number of material balance periods without detection. Eventually, perhaps, the systematically low values for transfers (42), (52) and (62) will lead to a low estimate of consumption due to corrosion, thus casting suspicion on the high unexplained losses, but a dangerous amount of X could be diverted before this comes about.

The recovery and decontamination operations should, therefore, be closely guarded to prevent diversion. Equipment should be processed quickly, with material in a form liable to diversion as short a time as possible. As noted on page 44, if detailed data are kept by the decontamination and recovery departments, it will be possible to predict fairly well the amount of T to be expected from the particular collection of equipment sent to recovery operations during a particular material balance period.

It should also be noted that any material in any part of the plant which has dropped from sight of the accounting system may be diverted without detection by material accounting.

Because judgments about the probably magnitude of consumption and the possible existence of diversion depend so largely on the precision of estimates of "unexplained " and unaccounted losses, a statistical analysis of all data used in the

[redacted]

[redacted]

Estimates is essential. The estimates for the unaccounted losses during a material balance period involve small differences between large amounts of material crossing the material balance envelope. The precision of the measurements of these streams and the initial and final inventories is critical if estimates of the unaccounted losses are to have adequate precision.

The next section of this report deals with estimates of the precision with which losses may be estimated.

[redacted]

VII. PRECISION AND ACCURACY OF MAIN PROCESS MATERIAL BALANCE

1. Introduction

Questions of precision are often considered at the end of an engineering study to judge the reliability of conclusions, and to indicate the range of applicability of results. In the present case, however, considerations of precision (and accuracy) will be decisive in outlining the inventory and material balance procedures. So many quantities have to be measured that careful consideration must be given to each so that no more effort than is needed is spent on unimportant items, and so that sufficient care is expended on decisive items. It is difficult indeed to judge beforehand just which items are likely to contribute most to uncertainty but a method is offered that will show what work has to be done to determine any inventory, (or the overall material balance on T or X) with any stated precision. It will also be seen how and where to allocate further efforts, if increased precision is desired. For certain ranges of precision, energy must be expended on measuring certain streams and the inventories of certain vessels. As the precision-requirements are made more stringent, the attention shifts to different streams and vessels, since no considerable further increase in precision is possible by more precise measurements on the set concentrated on at first.

Three sets of concepts are assembled for designing the material balance procedure. In the first place it is necessary to write down an analytical expression for the overall material

[redacted]

[redacted]

balance in terms of measurable quantities. This part of the work is familiar to engineers but is usually not extended to the degree necessary at K-25. The value of the product, the risks of loss or diversion of U-235, and the extensiveness of the plant require a full listing in this case.

In the second place, it is necessary to have an estimate of the precision with which each measured quantity entering the material balance equation is known. In the field of statistics, the idea of precision has been refined in recent years so that statements can be made of how precisely we know a quantity that are quite as objective as the measurements themselves. The use of confidence-belts makes it possible to say how certain we are of the numerical conclusions drawn. Thus, when it is specified that we wish to be 95 per cent certain that, say, the assay of a waste drum is within $\pm$ 0.1 per cent of the reported value, it is possible to state how many measurements must be made in order to assure this precision.

In the third place, it is necessary to have a set of equations showing how the precision of a derived quantity, such as the T-inventory, is related to the precision of each of the physical quantities on which it depends. Such a set of equations, originally developed by Gauss, are called the propagation-of-error equations. These relations show how the small random errors always made in the measurement of physical quantities affect the magnitude of derived quantities.

[redacted]

[redacted]

This section of the memorandum is principally concerned with the effect of all measurement errors on the uncertainty in the values of the unaccounted losses of T and X. The statistical analysis in this section is limited to the K-25 plant alone, but the results are a valid approximation for combined operation of the K-25 and K-27 plants. The present analysis in many cases relies on guesses as to the precisions of individual measurements. It is recommended that the analysis be extended to combined operation as soon as fairly complete information is available concerning the precisions of individual measurements.

The unaccounted for loss of T (E_T) within the main material balance envelope of Figure I is given by

$$
\begin{aligned}
E_T = & \sum (\text{T - Streams Entering Envelope}) \\
& - \sum (\text{T - Streams Leaving Envelope}) \\
& + (\text{Inventory within Envelope at start of material balance period}) \\
& - (\text{Inventory within Envelope at end of material balance period})
\end{aligned}
$$

A similar expression gives the unaccounted loss of X (E_X).

The certainty with which the losses E_T and E_X can be determined depends on the precision and accuracy of all stream and inventory measurements.

The analysis proceeds by stages, starting with relatively simple situations:

In Parts 2 and 3 the concepts of precision and accuracy are discussed, and the propagation-of-error equation introduced.

In parts 4 and 5 we find the precision with which the

[redacted]

T-inventory of Sections 300 and 600 may be determined. It turns out that an individual inventory determination has a confidence belt of ±23 kgm., and that the difference between two successive inventories (the quantity of importance in the material balance) has a confidence belt of ±32 kilograms. This places a lower limit on the precision with which E_T can be determined. The lack of precision due to [illegible] stream measurements must be added to this quantity to obtain the overall precision of E_T.

In Part 6 the effect of <u>bias</u> in the Section 300 and 600 inventory determination is discussed. If the equipment volumes used in calculating inventories from pressure readings are in error, the difference between two inventories may be in error, increasing the ±32 kgm. figure given above to as much as ±34 kgm. in an unfavorable case.

In Part 7 the uncertainty in E_T due to lack of precision of the stream measurements is discussed.

In Part 8 it is shown that for material accounting purposes, T-stream measurements may be regarded as unbiased.

The total uncertainty in E_T resulting from imprecision and bias in T-inventory, and imprecision in T-stream measurements is given by Figures 1 and 2.

In Parts 9, 10 and 11, the above analysis on the T material balance is repeated for the X balance. It is found that a single determination of the Section 300 and 600 X-inventory

[redacted]

[redacted]

has a confidence belt of ±0.6 kgms., and that the difference between two successive inventories has a confidence belt of ±0.85 kgms. This is the lower limit on the precision with which E_X can be obtained.

The overall precision of the X material balance, including the contributions due to stream measurements, is given by the lower curves of Figures 3 and 4.

In Parts 12 and 13 a serious limitation on the degree of certainty of the X balance is examined. The possible bias of the analytical methods used for product, feed, and waste assay contribute to the uncertainty of the X balance as shown by the upper curves of Figures 3 and 4: Curve I represents the current conditions of assay accuracy; Curve III represents what is believed to be an attainable goal.

In Part 14 the measurement of minor inventories and streams is discussed. A criterion for dumping of carbon and aluminum traps is presented.

In Part 15 the problems involved in localizing plant consumption of T and X are briefly indicated.

In Part 16, an overall material balance on U-234 is outlined. This is a further check on T consumption.

2. Precision

It has long been the custom of physicists and engineers to improve the certainty of their measurements of a quantity by taking repeated measurements. Everyone feels that the average of several independent measures of the same physical situation

[redacted]

[redacted]

is more precise than a single measure. It is generally the case that, as more and more measurements are made, the average of all the measurements becomes steadier and steadier. The reason for this improved stability lies in the way in which those causes of deviations which are random, balance each other out. As more and more measurements are made, the probability of random errors balancing increases, and thus the average value will show their effect less and less. The average thus converges into a decreasing range as the number of independent measures increases.

This type of convergence does not always occur, however, and the reasons for this failure are to be found in the non-applicability of certain assumptions implicit in the situation outlined above. In the first place the measurements may not be statistically independent. This is the case when there is correlation of any sort between successive measurements. If the measuring device wears down, or "remembers" previous measurements, then the assumption of statistical independence is not valid. The other cause of the failure of repeated measurements to converge on a single value is to be found in the quantity being measured. If the external controlling conditions are shifting so that actually one is not measuring the same quantity, then of course one cannot hope for convergence to a stable average. In the statistician's terms, one is not sampling from the same population. Evidence on this point can usually be obtained from data themselves, if a long series of measurements is made.

[redacted]

[redacted]ET

It will be assumed in this discussion that the (infinite) population is stable and, further, that the measurements to be made are statistically independent so that convergence to a stable average can be expected. The average obtained from a sequence of, say ten measurements will of course not be the same as the average of a second set of ten measurements, because the random errors cannot be expected to balance each other in the same way in two small samples. However, if one can determine the variability of single measures, a simple relation exists between this quantity and the variability of the average of N measurements. The best measure of variability is the <u>standard deviation.</u> This is defined by the equation:

$$s(x) = \sqrt{\frac{\sum_i (x_i - \bar{x})^2}{N - 1}} \qquad (1)$$

x_i	=	A single measurement
$\bar{x}$	=	the average of N measurements
$\sum_i$	=	represents a sum of all values of $(x_i - \bar{x})^2$
$s(x)$	=	the standard deviation of single measurements, an estimate of the variability existing in the (infinite) population of possible measures, based on a "sample of size N".

Imagine now that a large number of samples of size N are drawn from the same population, i.e., a series of sets of N measurements are made, and an average ($\bar{x}$) is computed from each set. The values of $\bar{x}$ will not all be the same. They will in fact vary around <u>their</u> average ($\bar{\bar{x}}$) and it will be possible to compute a standard deviation of these averages, by using Equation 1.

[redacted]

[redacted]

However, a very large amount of data would have to be collected for this purpose and it is often preferable to use the relation shown in Equation 2 instead, which relates the standard deviation of <u>single</u> measurements around their mean, $s(x)$, to the <u>standard deviation of a mean</u>, $s(\bar{x})$, (often called the <u>standard error of the mean</u>).

$$s(\bar{x}) = \frac{s(x)}{\sqrt{N}} \qquad (2)$$

The derivation of this equation does not involve any assumptions as to the frequency-distribution of the original values. A simple derivation is given in Croxton & Cowden, Applied General Statistics, Appendix E, Section XII-1. It is worth mentioning that the distribution of <u>means</u> of even quite small samples (N=4) follows the so-called normal or Gaussian distribution, even though the parent population is of widely different form.

The square of the standard deviation is called the <u>variance</u>:

$$V(x) = s^2(x) \qquad (3)$$

It is used because most relations between two or more variabilities are linear in V and not in s. In fact, if a quantity A is known to be equal to the sum or difference of two measured quantities B and C and if the random small errors in B are independent of those in C, it can be shown that

$$V(A) = V(B) + V(C) \qquad (4)$$

[redacted]

[redacted]

Proof of this relation, which does not depend on the form of the distribution of errors in B and in C, is given in Appendix I.

Since neither the standard deviation nor the variance have a ready physical interpretation, it will generally be more convenient to summarize results in terms of a "95 per cent confidence-belt", which is the range inside which the mean value will be expected to lie in 95% of a large number of repetitions of the taking of a sample of N measurements. It is defined by the relation

$$d(0.05,\bar{x}) = \pm\, t(0.05,N-1) \cdot s(\bar{x}) \qquad (5)$$

where: $d(0.05, \bar{x})$ = 95% confidence belt for the mean of a sample of N measurements.

$t(0.05,N-1)$ = "Student's" t. The range, in units of sample standard deviations, inside which the true mean may be expected to lie, with 95% certainty" (see any text on statistics). For N > 20, t may conveniently be replaced by the value 2. For small N, t increases rapidly.

$s(\bar{x})$ = Standard error of sample average.

The quantity $d(0.05,\bar{x})$ is more satisfactory to remember than the variance, since it has the same dimensions as the quantity measured, and makes assertions of uniform certainty about variability. (The variance is generally used in computation since it follows simpler algebraic rules.)

[redacted]

[redacted]

The variability discussed above (due to small random errors), measured by a standard deviation or a variance or a confidence belt, is referred to as Precision. This is a little unfortunate, since imprecision would describe it better. In the remainder of this section, the concept of precision will be applied to the determination of the precision of the overall material balance, in particular, the precision with which one can state a value for the unaccounted losses of T and X.

3. Accuracy (Systematic Error)

We now make the distinction between precision and another form of error, which measures Accuracy. If it happens that the average of successively larger numbers of independent measures converges to a value which is known on other grounds to be an incorrect one, then it is clear that there must be some systematic error in the measurements (or in what they are supposed to be measures of). Systematic error, also called bias, cannot be handled by the methods indicated above. If, however, rough estimates of the magnitude of systematic errors in several averages can be obtained, then a (consistent) estimate of the magnitude of the error in any derived quantity can be made. Methods for doing this will be discussed later in connection with each of the measured quantities which may be subject to systematic error. It is essential that some independent information be available for an average value to be judged biased.

It may well turn out that the material balances proposed below will give indications that some averages are biased since the material balance is itself an independent physical check

[redacted]

[redacted]

on a necessary relation between all the quantities measured. The consumption of T and X in the K-25 plant may roughly be estimated from the corrosion characteristics of the materials of construction. These estimates should improve as more corrosion data become available. If the values of the unaccounted losses of T and X determined by the material balance turn out to be negative or to be considerably larger than previously estimated values, and if the precisions with which they are determined do not admit the previously estimated values as probable values, one may suspect that some of the measured quantities entering into the material balance are biased. If, however, reasonable values for the unaccounted losses of T and X are secured, the material balance furnishes no usable information on the bias of the individual measurements. Hence, we may secure evidence of bias, but not of lack of bias, from the results of the material balance.

[redacted]

4. <u>Precision of T-Inventory of the Cells of Section 300 caused by Error in Tails Pressure.</u>

The typical cell on-stream in Section 300 is a rather complicated hydrodynamic system, handling a gas mixture largely consisting of C-616 and G-74 of variable composition and pressure. The C-616 inventory of a cell is a function of each stage's operating tails pressure, temperature, concentration of G-74 and other diluents, and barrier permeability. The inventory also depends very slightly on the cell's environment (e.g., the permeability of the stages just above and just below the cell in the cascade, and the line pressure drops between the cell and these neighboring stages). The following measurements are available for the purpose of estimating a cell's TF_6 inventory:

1. Building datum pressure.
2. Six stage tails pressure measurements (actually the difference between the tails pressure and the building datum pressure is measured).
3. Six control valve angle measurements, which indicate the stage permeabilities. (Strictly, a control valve angle at a given stage is largely determined by the permeability of the stage below. Hence, the six control valve angle measurements of a cell reflect the permeability of Stages 1-5 of the cell in question, and of Stage 6 of the cell below. However, in evaluating the inventory of a large number of cells, the correct average permeability is obtained, and hence the correct average inventory insofar as inventory depends on permeability.)

[redacted]

[redacted]

4. Six stage temperature measurements

5. One G-74, oxygen, and C-816 concentration measurement (in the light stream of Stage 6).

A detailed procedure for calculating the inventory of Sections 300 and 400 will be presented in a future memorandum from the Engineering Development Division. These methods are too complicated for convenient use in the present statistical study of the propagation-of-errors in the overall material balance, and so simpler approximate methods will be used here. The results of the statistical study will not be vitiated by their use. It is emphasized that the equations and numerical constants given in this report are not recommended for use in actual inventory calculations.

The following equation will be used in this memorandum to estimate the T-inventory of a Section 300 cell:

$$T = A^{\circ}\bar{P}[1 - b(\Delta\bar{CV})][1 - d(\%N_2)][1 - e(\%816)] \times [1 - g(\%O_2)][\quad /\bar{R}] \qquad (6)$$

where T = T-inventory of cell in kilograms

A° = inventory of cell operating with pure TF_6, and with all stages having a standard permeability, i.e., all control valves having a standard position. A° will be termed the cell "size factor".

$\bar{P}$ = mean tails pressure in psia

$\bar{CV}$ = mean difference between observed control valve position and standard control valve position.

$\%N_2$ = mol per cent nitrogen in sixth stage A pump discharge

$\bar{R}$ = mean stage temperature, $^{\circ}R$.

[redacted]

[redacted]

Values for the factors b and d have been derived from "Plant Inventory as a Function of Control Valve Position and Nitrogen Concentration", Report 2.16.2, by F. Zenz and E. Welsh, 12-3-45, and are given in Table XI of Appendix B. The factor d is less than 0.01 because the mean concentration of nitrogen in a cell is less than that in the light stream. The inventory should be corrected for other diluents in a similar manner to the correction for nitrogen ("d" for oxygen is taken as equal to d; an l-value of 0.01 is used for C-816.)

The following table shows which quantities in Equation (6) affect the precision of an inventory determination, and which affect its accuracy:

Variables which may be measured as often as desired in order to increase precision of inventory	Parameters which remain constant except when equipment is recalibrated or new computing procedures are introduced. Errors in these parameters render an inventory inaccurate
$\overline{P}$	
$\overline{CV}$	A^o
$\% N_2$	[illegible]
% 816	d
$\% O_2$	l
$\overline{R}$	q

We now consider the precision with which the T-inventory of Section 300 cells can be determined from measurements of the variables appearing in Equation 6. For simplicity in exposition we will first assume that all stages have a standard permeability, 0.0% N_2, C-816, and O_2, and operating temperature, so that Equation (6) simplifies to

$$T = A^o \overline{P} \tag{7}$$

[redacted]

We will first calculate the contribution of the precision of the tails pressure measurement to the precision of the inventory determination, and later add the contribution of control valve position, diluent concentrations, and stage temperature measurements.

Our goal will be to state the precision with which the inventory can be determined, and the contribution of each variable to this precision (in order that work to increase precision may be concentrated on the more important variables). We will also be able to state the smallest difference between two T-Inventory determinations which we may determine with confidence. This limiting difference will be of importance when we consider the overall material balance on T.

The following equation gives the T-inventory of all cells of Sections 300 under the assumptions used in writing Equation 7:

$$T_{cells} = \sum_{i=-3}^{4} A_i^o \, n_i \overline{P}_i \qquad (8)$$

where

T_{cells}	=	inventory of T in the cells of Section 300, uncorrected for nitrogen, oxygen, 816, and permeability. (kgms.)
A_i^o	=	size factor for the cells of Section i (i running from Section -3 through Section 4). kgms./psig.
n_i	=	number of active cells of Section i
$\overline{P}_i$	=	Weighted average tails pressure of all stages of Section i. psig.

[redacted]

[redacted]

It will be noticed that Equation 8 is a sum of products. As is shown in Appendix I, when $F = G \times H$, the variance of the product, $V(F)$, is related to the variance of the factors $V(G)$ and $V(H)$ by the relation:

$$\frac{V(F)}{F^2} = \frac{V(G)}{G^2} + \frac{V(H)}{H^2} \tag{9}$$

or

$$V(F) = F^2 \left[V(G)/G^2 + V(H)/H^2 \right] \tag{10}$$

$$= H^2 V(G) + G^2 V(H)$$

and similarly for the product of any number of factors. No assumption is made about the frequency-distribution of errors in A and B, but statistical independence is required and the errors in each quantity must be small with respect to the quantity itself.

Applying Equation 9 to 8,

$$V(T_{cells}) = \sum_{i=1}^{4} T_i^2 \left(\frac{V(n_i)}{n_i^2} + \frac{V(\bar{P}_i)}{\bar{P}_i^2} \right) \tag{11}$$

where T_i = T-inventory of all cells in Section i.

$V(n_i)$ will be set equal to zero, since we cannot afford an error as large as a one cell miscount. Furthermore, it seems reasonable to assume that the uncertainty of pressure will be the same in all sizes of equipment. Let $V(P)$ be the variance of a large number of individual tails pressure measurements. Then the variance of the average pressure of a section is

$$V(\bar{P}_i) = \frac{V(P)}{6n_i} \tag{12}$$

by Equation 2, since there are 6 n_i tails pressure measurements in Section i. Equation (11) becomes:

[redacted]

[redacted]

$$V(T_{cells}) = \sum_{i=-3}^{4} \frac{V(P)}{6\,n_i}\,\frac{T_i^2}{\bar{P}_i^2} \qquad = \frac{V(P)}{6}\sum_{i=-3}^{4} A_i^2\, n_i \qquad (13)$$

The quantities A_i^o and n_i are roughly known, but a rough estimate must now be made of V(P). This variance depends on the variability of the individual tails pressure recorders and the reliability of the building datum pressure recorder. If we assume that 95 per cent of the recorded tails pressures are within ±0.1 of the scheduled values, then

$$d(05,P) \doteq \pm 0.1 \text{ psia}$$
$$= t \times s(P) = 2\sqrt{V(P)}$$

The factor 2 is used as an approximation for t.

$$V(P) = \overline{0.05}^{\,2} = 0.0025 \text{ (psia)}^2$$

Table II shows the estimates of A_i^o and n_i used:

Table II

Variance of T Inventory Due to Randon Error in Pressure Measurements --

Section 300 cells

Section	Appro ximate Values of Cell Size Factor A_i	Number of Cells in Section i n_i	Variance of Pressure Reading V(P)	Variance of T-Inventory of Section $\frac{V(P)}{6} A_i^2 n_i$
-3		9	0.0025	
-2		23	0.0025	
-1		15	0.0025	
1		48	0.0025	
2		127	0.0025	
3		164	0.0025	
4		94	0.0025	
Total		480		54.1

[redacted]

[redacted]

Therefore $s(T_{cells}) = \sqrt{56.1} = 7.35$ kgms.

and $d(05, T_{cells}) = \pm 14.7$ kilograms

$=$ per cent

This calculation shows that, if all pressures are recorded and averaged, the inventory of T in the Section 300 cells may be computed to about per cent. Since the size factors A_i^o are almost certainly biased due to errors in their estimation, we have here a case of convergence on a biased average, but since we will be concerned in the future with changes in inventory, this bias will largely disappear. The effect of this bias on the material balance is calculated in part 6.

The uncertainty in the difference between two T-inventories may be computed using Equation 4. Let T^{I}_{cells} and T^{II}_{cells} be two successive determinations of the Section 300 cell inventory.

Then
$$V(T^{I}_{cells} - T^{II}_{cells}) = V(\Delta T_{cells}) = V(T^{I}_{cells}) + V(T^{II}_{cells})$$
$$= 2V(T_{cells}) \quad (14)$$
$$= 2 \times 54$$
$$= 108$$

$\therefore d(0.05, T_{cells}) = 2\sqrt{108} = \pm 20.8$ kgm.

Thus if all cells contain pure TF_6, and are of the same permeability and temperature, a change in T-inventory of Section 300 cells cannot be detected, with 95 per cent certainty, if it is less than 21 kilograms. It would be premature to use this value, however, since the factors discussed below increase this value considerably.

[redacted]

[redacted]

5. Precision of T-Inventory of Sections 300 and 600

The preceding discussion has let to an estimate of the precision of a determination of the T-inventory of Section 300 cells omitting the contributions to uncertainty due to random errors in the estimates of cell permeability, temperature, $\%N_2$, $\%316$, and $\%O_2$ measurements. Appendix B gives an analysis of these omitted measurements. Appendix C shows the additional uncertainty added by errors of measurement in piping, and in the T-inventory of Sections 600 and 312. These uncertainties, expressed as variances, may be added to the variance found in part 4 to obtain the total uncertainty from all causes. Table III lists all contributions of uncertainty to the T-inventory thus far examined.

[redacted]

TABLE III

Summary of Causes and Magnitudes of
Random Error in T-Inventory
Sections 300 and 600

Variable Measured	Number of Measurements	95% Confidence Belt on each Measurement	Variance $(\text{kgm.T})^2$
Cell Tails Pressure	2,900	±0.1 p.s.i.a.	54.1
Cell Permeability	2,900	±5 % closed	53.1
Cell Temperature	2,900	±5 °F	1.3
Cell $\%N_2$	100	±0.10 x ($\%N_2$)	0.0
Cell % C-816	36	±0.10 x (%C-816)	2.0
Intercell Piping pressure	475	±0.1 p.s.i.a.	4.7
Interbuilding Piping Pressure	51	±0.1 p.s.i.a.	1.7
600 Section Surge Drum Pressure	1	±0.05 p.s.i.a.	9.8
600 Section Surge Drum Temperature	1	±5 °F	0.0
312 Section Pressures	40	±0.1 p.s.i.a.	0.0
Total			126.7

[redacted]

Although it is hoped that the terms due to tails pressures, and to Section 600 will be considerably reduced in the near future, the values listed in Table III will be used in this report in order to be conservative.

It is clear from the value given for total variance that a single T-inventory can be expected to be precise to within $2\sqrt{130} = \pm 23$ kgms. A difference between two inventories will, then, be precise to within $\pm 23\sqrt{2} = \pm 32$ kgms.

The possibility of reducing the effect of some of the larger sources of error can now be considered. Looking at Table III, the items due to pressure-uncertainty, to control-valve-angle-uncertainty, and to Section 600 inventory uncertainty seem most pormising.

The uncertainty due to pressure is estimated from a rather pessimistic view of the confidence-belt on single pressure readings. A study of the actual current conditions is now underway. This study will probably give a lower 95% confidence-belt on pressure than 0.1 p.s.i.a. and will, further, probably give indications of the amount of work involved in improving the calibration of the pressure recorders. It will then be possible to judge the feasibility of improving the precision of the T-inventory by reducing d(0.05,P).

[redacted]

[redacted]

Uncertainty in control-valve-angle indication is also under study, in the hope that the current estimate $d(0.05, CV) \approx \pm 5\%$ will prove to be high, and also that improved calibration will further reduce the value found.

It will be noticed in Table XI(a), Appendix B, that the confidence belt on control valve angle is taken to be the same in all Sections of the plant. This is certainly not the case for Section 4, but even a large increase in the value of V(CV) for this section will not influence the precision of the cascade T-inventory to any large degree since the weighting factor T_4^2 is so small for this section.

Methods for finding the pressure and temperature of Section 600 are also under study. It will probably not be difficult to reduce the term 9.8 kgms.^{2}, given in Table III and in Appendix C, to a value near 2.0 kgms.^{2}.

Since, therefore, all the major contributions to T-inventory uncertainty have probably been over-estimated, the value of 130 kgms.^{2} given above for the random-error-variance of T-inventory is felt to be conservatively high.

6. Accuracy of Section 300 T-Inventory

The discussion thus far has been concerned with precision. The question answered by a precision study is: If all the direct measurements are repeated with the same instruments and the same care, within what range will the derived quantity lie (with 95% certainty)? We now must guess how large a systematic error in

[redacted]

[redacted]

the T-inventory results from constant errors in the quantities measured. It will be found that a fairly large systematic bias in the individual T-inventories can be tolerated. Our main interest is in the difference between determinations of the cascade's T-inventory, since our estimate of the unaccounted-for loss of T depends on this difference.

Referring to Table III, we will consider the constant errors that may exist in the measurements listed. It does not seem reasonable to assume the existence of bias in the tails pressure, number of cells, control valve angles, concentration of contaminants, and temperature, since each of these quantities can be checked exactly or calibrated against standards of known accuracy if it is necessary to reduce their bias to a desired minimum. On the other hand, the cell size factors A_i^o, the coefficients used for estimating the effect of control valve angle and G-74 concentration on inventory, and the volumes of intercell and interbuilding piping and Section 500 may well be biased. To simplify the discussion, only the size factors will be considered.

The cell size factors A_i^o are each determined from a series of repeated calibration tests, and so a confidence belt will be available for each. Since it is planned to use the size factors determined from a single series of calibration tests for a number of inventories, any error in these size factors will affect

[redacted]

[redacted]

the results in a consistent way, instead of in a random way as is the case with errors in quantities which are redetermined at each inventory time. The confidence belts on the average size factors obtained from calibration tests are measures of the possible bias in the values of the size factors regularly used for inventory determinations. Of course, the calibration technique itself may be biased, but on this point we have no information.

The effect of the possible bias in size factors on the uncertainty of the difference between two inventories is determined by the propagation-of-error equation. The equation for the change in inventory should be written

$$\Delta T_c = \sum_{i=-3}^{4} A_i^\circ \, \Delta(n_i \bar{P}_i) \tag{15}$$

instead of

$$\Delta T_c = \sum_{i=-3}^{4} \Delta(A_i^\circ n_i P_i) \tag{16}$$

because the same values of A_i° are used in computing both inventories. The change in cascade inventory is represented by ΔTc. The variance expressing the uncertainty in the inventory change caused by bias should be written as

$$V_b(\Delta T_c) = \sum_{i=-3}^{4} \overline{\Delta(n_i \bar{P}_i)}^{\,2} V_b(A_i^\circ) + 2\sum_{i=-3}^{4} A_i^{\circ 2} V_b(n_i \bar{P}_i) \tag{17}$$

instead of

$$V_b(\Delta T_c) = 2\sum_{i=-3}^{4} (n_i P_i)^2 V_b(A_i^\circ) + 2\sum_{i=-3}^{4} A_i^{\circ 2} V_b(n_i \bar{P}_i) \tag{18}$$

[redacted]

[redacted]

where V_b has been used to denote variance due to bias. We agreed above that the bias in n_i and $\overline{P}_i$ must be negligible, and so only the first term of (17) remains. The quantity $V_b(A_i^o)$ may be estimated from the confidence belt on the cell inventory calibrations.

Let us assume that in the worst case the tails pressures may be changed by 10% throughout Section 300. It will further be assumed that each A_i^o is the average of six calibrations, and that the 95% confidence belts are ±3%, ±3%, ±3% and ±5% respectively for the four equipment sizes. Then

$$\Delta(n_i P_i) = 0.1\, n_i \overline{P}_i$$

Sections -1, 1

$$V_b(A_1^o) =$$

Sections -2, 2a, 2b

$$V_b(A_2^o) = ($$

Sections -3, 3a, 3b

$$V_b(A_3^o) = ($$

Section 4

$$V_b(A_4^o) =$$

$$t(0.05, 5) = 2.45$$

[redacted]

[redacted]

With these assumptions a numerical estimate of the probable bias in ΔT_c can be made. Table IV shows the computation. It is included so that the relative contributions of the different factors and terms may be seen

TABLE IV

Estimation of possible Variance in Change in Cascade T-Inventory Due to Bias in Cell Size Factors

Section	Number of cells in Section n_i	Mean Tails Pressure in Section P_i	$(0.1\ n_iP_i)^2$	Variance of Size Factor, $V_b(A^o_i)$	Variance of change in inventory, $V_b(\Delta T_i)$
-3	9				
-2	23				
-1	15				
1	37				
2a	45				
2b	89				
3a	48				
3b	118				
4	89				
				Total	30.3

It is suggested that this quantity be added by the usual propagation-of-error method to the uncertainty in ΔT due to random errors. Thus the total uncertainty in ΔT_c may be written as

[redacted]

[redacted]

$$d(0.05, \Delta T_c) = 2\sqrt{260+30} = \pm 34 \text{ kgm. } T$$

or $V(\Delta T_c) = 290 \text{ kgm}^2 \, T.$ This value for $V(\Delta T_c)$ will be used in Part 7, in determining the uncertainty of the material balance on T.

If an overall material balance on T is run during a period of changing T-inventory, it will be possible to use this fact to estimate systematic errors in the A_i^o. The pressures need only be raised (or lowered) section by section, with a cascade inventory taken before and after each pressure change. The change of inventory can then be used to estimate the size factor for each section with good precision and with greatly improved accuracy.

The error in ΔT_c introduced by using the same A_i^o in both inventories can only be reduced by making more, or more careful measurements of the A_i^o. The probable bias in ΔT_c will be reduced proportionally to the fractional reduction in the $d(0.05, \overline{A_i})$, if these are everywhere the same. However, as can be seen from Table III, almost all the bias is transmitted through A_1 and A_2, so that these two should be re-calibrated if more <u>accuracy</u> is required in the change in T-inventory.

It will be shown later (Part 10) that A_3^o and A_4^o are of most importance in improving the <u>accuracy</u> of the X-inventory.

[redacted]T

7. Precision of Material Balance on T

The overall T-balance for a material balance period is given by the equation

$$E_T = T_F - T_W - T_\Pi - \Delta T_c = n\,e_T \qquad (19)$$

where

E_T = Overall unaccounted loss of T during material balance period

T_F = kgms. T in feed stream, throughout the balance period

T_W = kgms. T in waste stream

T_Π = kgms. T in product stream

T_c = Cascade T-inventory at end of period less that at beginning, i.e., the increase in T-inventory

n = number of days in material balance period

e_T = average daily rate of unaccounted loss of T

This equation omits minor inventories and minor streams crossing the main process material balance envelope. These will be discussed in Part 13, and the precisions of their measurements will be specified so that they make a minor contribution to the overall uncertainty of the T-balance.

The random-error equation corresponding to Equation (19) is

$$V(E_T) = V(T_F) + V(T_W) + V(T_\Pi) + V(\Delta T_c) \qquad (20)$$

$$= \sum_1^{N_F} V(\bar{F}) + \sum_1^{N_W} V(\bar{W}) + \sum_1^{N_\Pi} V(\bar{\Pi}) + V(\Delta T_c) \qquad (21)$$

[redacted]T

[redacted]

where N_F = number of feed drums used

N_W = number of waste drums used

N_Π = number of product drums used

$\bar{F}$ = estimated weight of T in a single feed drum *

$\bar{W}$ = estimated weight of T in a single waste drum*

$\bar{\Pi}$ = estimated weight of T in a single product drum*

In the last part of this section, a value of 290 $kgm.^2$ was found for $V(\Delta T_c)$. This is seen to be a lower limiting value for $V(E_T)$. We will now derive expressions for $V(\bar{F})$, $V(\bar{W})$ and $V(\bar{\Pi})$ so that their contributions to $V(E_T)$ can be evaluated.

The weight, w, of TF_6 in each drum is found by the difference between the weight full, w_1, and the weight empty, w_2.

The precisions expressed as 95% confidence belts of the feed, waste and product scales are all roughly 0.25 per cent of the amount weighed.

$$w = w_1 - w_2$$

$$V(w) = V(w_1) + V(w_2)$$

$$V(w_1) = \frac{1}{n_{w_1}} \left(\frac{0.0025\, w_1}{2 \times 2.2} \cdot \frac{238}{352} \right)^2$$

$$V(w_2) = \frac{1}{n_{w_2}} \left(\frac{0.0025\, w_2}{2 \times 2.2} \cdot \frac{238}{352} \right)^2$$

$$V(w) = 14.5 \times 10^{-8} \left(\frac{w_1^2}{n_{w_1}} + \frac{w_2^2}{n_{w_2}} \right) (kgm.\ T)^2 \tag{22}$$

* It is assumed in this part that the chemical purity of feed product and waste streams is known with full precision. The effect of random error in estimating the chemical purity of streams is discussed in Appendix D and immediately following Table V in this part.

[redacted]

[redacted]

where w_1 and w_2 are in lbs. of TF_6. In these equations, n_{W1} is the number of independent weighings of each full feed drum and n_{W2} is the number of weighings of each empty drum. For the feed drums w_1 is about 650 pounds and w_2 about 150 pounds. For feed weighings, then

$$V(\bar{F}) = 14.5 \times 10^{-8} \left(\frac{\overline{650}^2}{n_{w1}} + \frac{\overline{150}^2}{n_{w2}} \right)$$

$$= 0.061 \left(\frac{1}{n_{w1}} + \frac{0.053}{n_{w2}} \right) \qquad (23)$$

It is clear from Equation (23) that most of the uncertainty in weight of T per drum comes from the uncertainty in weighing the full drum. There will then usually be little improvement by repeated weighings of the empty drum, unless the full drum has already been weighed then or more times. Let us recommend that the empty drum be weighed once for each 5 weighings of the full drum, but in no case should the empty drum be weighed less than twice to avoid errors due to carelessness. For this schedule, Equation (23) becomes

$$V(\bar{F}) = \frac{0.077}{n_F} \qquad (24)$$

where n_F is the number of repeated independent weighings of each full feed drum. (Equation (24) does not strictly apply where

[redacted]

[redacted]

n_F is below 10, due to our requirement that the empty drum be weighed at least twice.

Since the full waste drum weighs about 6500 pounds, and the empty drum about 1500 pounds. Equation (22), applied to the waste uncertainty becomes

$$V(\overline{W}) = \frac{7.7}{n_W} \qquad (25)$$

where n_W is the number of independent weighings of each full waste drum, under the same weighing schedule as for feed drums.

The corresponding equation for product is:

$$V(\overline{\Pi}) \doteq \frac{0.00002}{n_\Pi} \qquad (26)$$

since the full and empty product drums weigh about 10 and 7 pounds, respectively. It is obvious that uncertainty in weight of product makes no significant contribution to uncertainty in consumption. This term will therefore be omitted below.

Substituting Equations 24 and 25, into Equation 21, noting that $N_F = 10\ n$, $N_W = n$, and that $V(\Delta T_0) = 290$ (from Parts 5 and 6):

$$V(ne_T) = n\left[\frac{10 \times 0.077}{n_F} + \frac{7.7}{n_W}\right] + 290$$

$$= n^2 V(e_T)$$

$$\therefore \quad V(e_T) = \frac{0.77}{n}\left(\frac{1}{n_F} + \frac{10}{n_W} + \frac{380}{n}\right) \qquad (27)$$

[redacted]

[redacted]

Letting $d(0.05, e_T)$ represent the 95% confidence belt on e_T:

$$d(0.05, e_T) = 2\sqrt{V(e_T)}$$

or

$$d(0.05, e_T) = \pm 1.75\sqrt{\frac{1}{n}\left(\frac{1}{n_F} + \frac{10}{n_W} + \frac{380}{n}\right)} \qquad (28)$$

This equation will repay some study, since it shows the relative importance of the three factors left at our disposal after the inventory procedure has been fixed. In the first place it is seen that repeated weighings of each waste drum reduce the uncertainty in e_T more than repeated weighings of each feed drum. Secondly, the relative importance of re-weighing, as against waiting a longer time can be judged by the relative magnitude of the two quantities $(1/n_F + 10/n_W)$ and $380/n$.

Since it will be difficult to weigh each waste drum much oftener than 10 times, let us tentatively put $n_F = 2$, and $n_W = 10$ in Equation (28)

$$d_1(0.05, e_T) = \frac{2.1}{n}\sqrt{n + 250} \qquad (29a)$$

$$d_1(0.05, E_T) = 2.1\sqrt{n + 250}$$

To compute the precision possible without changing current practice of weighing each feed and waste drum once, we place $n_F = n_W = 1$, obtaining

[redacted]

$$d_2(0.05, e_r) = \frac{5.8}{n}\sqrt{n+35} \qquad (29b)$$

$$d_2(0.05, E_r) = 5.8\sqrt{n+35}$$

Values of the precision under these two sets of assumptions concerning frequency of weighings are given in Table V

TABLE V

Precision of Estimate of Unaccounted Loss of T, as It Depends on Number of Feed Drum Weighings (n_F), on number of Waste Drum Weighings (n_W) and on Number of Days between Inventories (n)

Days between Inventories	95% Confidence Belt on Total Loss of T, kgms.T		95% Confidence Belt on Daily Average Loss T, kgms. T/day	
n	n_F 2, n_W 10	1, 1	2, 10	1, 1
5	±34	±37	±6.7	±7.3
10	34	39	3.4	3.9
20	35	43	1.7	2.2
30	35	47	1.2	1.5
60	37	56	0.62	0.94
90	39	65	0.43	0.72
120	40	72	0.34	0.60
180	44	85	0.24	0.47
240	49	96	0.20	0.40
360	52	115	0.14	0.32

[redacted]

[redacted]

The values for $n_F = 2$ and $n_W = 10$ are plotted in Figures 1 and 2.

It is clear that there is no great improvement in precision of e_T by repeated weighings if the material balance is for a period of time less than a month. If, however, longer periods of time are considered, say 6 months, then it becomes possible to double the precision by weighing feed and waste drums oftener as shown.

It is hardly necessary to remind the reader that the numerical results given in Table V are subject to a considerable number of assumptions. These assumptions are listed in Table I (found in Section II).

In the work up to this point it has been assumed that the chemical purity of feed, waste and product streams are precisely known. In Appendix D it is shown that the increase in the T-balance uncertainty is negligible if the chemical purity of each feed drum is known to 0.2 per cent and the purity of each waste drum to 0.1 per cent.

8. Accuracy of T Stream Measurements

The elimination of bias in the feed and waste streams is particularly easy in the overall material balance on T since the measurements are all weighings. Bias caused by systematic error in scales can be reduced to almost any desired quantity by cross-checking of feed and waste scales. The same calibration weights can be used repeatedly every day on both scales and at the same time the precision of each scales can be checked.

[redacted]

[redacted]

9. Precision of X-Inventory of Sections 300 and 600

The X-inventory of the cascade is determined by measuring the X-concentration at a large number of points, as well as all the quantities indicated in Table III. The precision of the X-inventory will be estimated by exactly the same method as used in the T-inventory, with one important difference. The X-concentration (called x, weight-fraction, throughout this section) varies widely in the cascade, even within a single Section, and therefore cannot be averaged as were pressures and control-valve angles in the T-inventory. For this reason the analysis of precision of X-inventory must be carried through by buildings instead of by sections.

The X-inventory of the cells in Section 300 will be given by:

$$X_{cells} = \sum_{j=1}^{N_B} A_j n_j \bar{P}_j \bar{x}_j \qquad (30)$$

where X_{cells} = X-inventory of Section 300 cells

A_j = size factor for cells in the j th building, kgms T/psia/cell, corrected for permeability, temperature, $\%N_2$, and %C-816 variability.

n_j = number of cells in building j

$\bar{P}_j$ = mean tails pressure, in building j

$\bar{x}_j$ = mean weight-fraction X in building j

N_B = number of Buildings in cascade

[redacted]

[redacted]

The corresponding equation for the variance in X-inventory caused by random error in n_j, $\bar{P}_j$ and $\bar{x}$ is:

$$V(X_{cells}) = \sum_j V(A_j^\circ\, n_j\, \bar{P}_j\, \bar{x}_j)$$

$$= \sum_j X_j^2 \left(\frac{V(n_j)}{n_j^2} + \frac{V(\bar{P}_j)}{\bar{P}_j^2} + \frac{V(\bar{x}_j)}{\bar{x}_j^2} \right) \quad (31)$$

We will now give rough numerical estimates of the quantities in the parenthesis and proceed to evaluate $V(X_{cells})$. We will assume that

a. $V(n_j) = 0$

b. $V(\bar{P}_j) = \dfrac{0.0025}{n_{P_j}} = \dfrac{417}{n_j} \times 10^{-6}$

c. $\dfrac{V(\bar{x}_j)}{\bar{x}_j^2} = \dfrac{81 \times 10^{-6}}{n_{xj}}$

where n_{Pj} = number of pressure measurements (= $6n_j$) in Building j

n_{xj} = number of independent assays in Building j .

Assumption a. is made because we cannot afford an error of one cell in the numbers n_j. Assumption b. is the same as that assumed in the T-inventory analysis, equivalent to d(0.05,P) = $\pm$ 0.1 p.s.i.a. Assumption c. is based on data from "B" and "C" laboratories, and is equivalent to assuming that d(0.05,x) = $0.018\bar{x}$.

Substituting a, b, and c in Equation 31

$$V(X_{cells}) = 81 \times 10^{-6} \sum_{j=1}^{N_B} X_j^2 \left(\frac{5.1}{n_j \bar{P}_j^2} + \frac{1}{n_{xj}} \right) \quad (32)$$

[redacted]

Table XIII, Appendix E, gives numerical values to the variables in Equation 32, using December 12, 1946 values of X_j, the full cell numbers per building for n_j and 2 for n_{xj}, meaning that two samples are taken in each building.

Table XIV, Appendix E, summarizes the calculations shown in Table XIII by Sections. The total variance of X_{cells} due to uncertainty in assay measurements (two per building) and pressure measurements (one per stage) is 0.0328 kgm.^{2}, with each type of measurement contributing about the same amount of uncertainty. The former can be considerably reduced by taking more samples for assay in Sections 3b and 4.

The additional uncertainty introduced by piping, Sections 312 and 600, and by the other process variables, (Control valve angle, $\%N_2$, % C-816) are computed by exactly analogous methods. Table VI summarizes all these contributions. The total uncertainty in the cascade X-inventory, X_c, is, then, 0.1 kgms.^{2}, corresponding to a 95% confidence belt of ± 0.6 kgms.

To find the amount of uncertainty in X_c if only a single assay sample per building is taken, it is only necessary to double the variance due to X-assay (0.0258 in Table XI). The total variance would be 0.134 kgms.^{2} instead of 0.108 kgm.^{2}. This corresponds to a 95% confidence belt on X_c of $\pm 2\sqrt{0.134}$ or ± 0.73 kgms. for a single inventory. A difference in two X-inventories of less that $0.73\sqrt{2}$ or ± 1.03 kgms. X could not be detected with 95% certainty with this schedule of measurements.

[redacted]

[redacted]

TABLE VI *

Summary of Causes and Magnitudes of Random Errors

in X-Inventory

Sections 300 and 600
(Two Assay Samples per Building)

Cause	Variance
X-assay	0.0258
Cell Tails Pressure	0.0370
Control Valve Angle	0.0134
Cell Temperature	0.0003
Cell $\%N_2$	0.0146
Cell % C-816	0.0010
Intercell piping	0.0117
Interbuilding piping	0.0014
Section 600	0.0030
Section 312	0.0000
Total Variance $(Kgm.)^2$	0.1082

* See Table III for assumptions concerning precision of measurements of all quantities except assays.

[redacted]

[redacted]

10. Accuracy of X-Inventory

The only measurement in the X-inventory that seems likely to introduce systematic error is that of the size-factors A_i^o. Exactly as in the T-inventory, these average values, since they are used twice, are certain to introduce some bias. Table VII below, gives a rough estimate of the variance caused by this bias, using average X-concentrations for each section.

$$V_b(\Delta X_c) = \sum_{i=-3}^{4} V_b(\Delta X_i) = \sum_{i=-3}^{4} V_b(A_i)\left[\Delta n_i P_i \bar{x}_i\right]^2 \qquad (32a)$$

We assume that $n_i P_i \bar{\Xi}_i$ changes by 10% in each cascade Section. The same uncertainty in each A_i^o is assumed as in Table III, Part 6, for the T-inventory bias.

[redacted]

[redacted]

TABLE XII

Estimation of Possible Variance in Change of X-Inventory caused by Bias in Size Factors

Section	Mean Weight Fraction $X, \bar{x}_i$	$\left[0.1\ n_i P_i \bar{x}_i\right]^2$	Variance in Size Factor, $V_b(A_i^o)$	Variance in change in X-Inventory of Section $V_b(\Delta x_i)$ kgm.^{2} X
-3				
-2				
-1				
1				
2a				
2b				
3a				
3b				
4				
			Total	0.0203

Since the sign of this bias is quite unknown, it must be added as a variance, to the other random-error variance already computed. The total variance of ΔX_c will then be $2 \times 0.108 + 0.02$ and this quantity is the one used in the following part for the total variance of the change in X-inventory, $V(\Delta X_c)$.

[redacted]

11. Precision of Overall Material Balance on X

By analogy with Equation 19, for the overall material balance on T, we write

$$E_X = n e_x = X_F - X_W - X_\Pi - \Delta X_c \qquad (33)$$

where E_X = overall unaccounted for loss of X, during material balance period, kgm. X

e_x = daily rate of unaccounted for loss of X, kgm. per day.

X_F = kgms. X in feed stream

X_W = kgms. X in waste stream

X_Π = kgms. X in product stream

X_c = increase in X-inventory of Section 300 during material balance period

n = number of days in material balance period.

This equation omits minor inventories and streams. These will be discussed in Part 13.

Substituting measured quantities in Equation (33)

$$E_X = n e_x = \sum_1^{N_F} \bar{x} \bar{F} - \sum_1^{N_W} \bar{x}_W \bar{W} - \sum_1^{N_\Pi} \bar{x}_\Pi \bar{\Pi} - \Delta X_c \qquad (34)$$

[redacted]

[redacted]

where

N_F = number of feed drums fed

N_W = number of waste drums filled

N_Π = number of product drums withdrawn

$\bar{F}$ = kgms. T per feed drum

$\bar{W}$ = kgms. T per waste drum

$\bar{N}$ = kgms. T per product drum

$\bar{x}_F$ = estimated weight-fraction of X in feed

$\bar{x}_W$ = assayed weight-fraction of X in waste

$\bar{x}_\Pi$ = assayed weight-fraction of X product

It will be sufficiently precise for current purposes (but not, of course, in the actual material balance operation) to assume that all feed drums contain the same weight of T, and similarly for all waste drums and product drums. Equation (34) may be written:

$$e_x = \frac{1}{\Pi}\left\{\sum_1^{N_F} \bar{x}_F \bar{F} - \sum_1^{N_W} \bar{x}_W \bar{W} - \sum_1^{N_\Pi} x_\Pi \bar{\Pi} - \Delta X_c\right\} \quad (35)$$

The corresponding random-error equation is:

$$V(e_x) = \frac{1}{\Pi^2}\left\{\sum_1^{N_F} V(\bar{x}_F \bar{F}) + \sum_1^{N_W} V(\bar{x}_W \bar{W}) + \sum_1^{N_\Pi} V(\bar{x}_\Pi \bar{\Pi}) + V(\Delta X_c)\right\} \quad (36)$$

[redacted]

[redacted]

$$V(e_x) = \frac{1}{\Pi^2}\left\{N_F V(\bar{x}_F \bar{F}) + N_W V(\bar{x}_W \bar{W}) + N_\Pi V(\bar{x}_\Pi \bar{\Pi}) + V(\Delta X_c)\right. \quad (37)$$

Since about 10 feed drums, 1 waste drum, and 10 product drums are used daily, we have

$$V(e_x) = \frac{1}{\Pi}\left\{10[\bar{x}_F^2 V(\bar{F}) + \bar{F}^2 V(\bar{x}_F)] + [\bar{x}_W^2 V(\bar{W}) + \bar{W}^2 V(\bar{x}_W)]\right.$$

$$+ 10[\bar{x}_\Pi^2 V(\bar{\Pi}) + \bar{\Pi}^2 V(x_\Pi)] + \frac{1}{\Pi} V(\Delta X_c) \quad (38)$$

Rough numerical estimates are available for all the quantities on the right hand side of Equation (38):

$$\bar{F} = \frac{500}{2.2} \times \frac{238}{352} = 154 \text{ kgms}$$

$$\bar{W} = \frac{5000}{2.2} \times \frac{238}{352} = 1540 \text{ kgms}$$

$$= \frac{3}{2.2} \times \frac{238}{352} = 0.92 \text{ kgms}$$

$$\bar{x}_F = 0.0071$$

$$\bar{x}_W = 0.0055$$

$$\bar{x} = 0.28$$

$$V(\bar{x}_F) = \frac{V(x_F)}{n_{x_F}} = \frac{(0.009\ x_F)^2}{n_{x_F}} = 81 \times 10^{-6} \frac{\bar{x}_F^2}{n_{x_F}} = \frac{4.13 \times 10^{-9}}{n_{x_F}}$$

[redacted]

[redacted]

where $V(x_F)$ = variance of a single X-assay

n_{x_F} = number of assays per feed drum and

similarly

$$V(\bar{x}_W) = \frac{V(x_W)}{n_{xW}} = \frac{(0.009\ \bar{x}_W)^2}{n_{xW}} = \frac{2.46 \times 10^{-9}}{n_{xW}}$$

$$V(\bar{x}_n) = \frac{V(x_n)}{n_{x_n}} = \frac{(0.009\ x)^2}{n_{x_n}} = \frac{6.4 \times 10^{-6}}{n_{x_n}}$$

The quantities $V(\bar{F})$, $V(\bar{W})$ and $V(\bar{n})$, are given by Equations 24, 25 and 26.

$V(\Delta X_0)$ has been estimated in the preceding part of this section as 0.23.

Substituting these twelve quantities in Equation (38):

$$V(e_x) = \frac{10}{n}\left[154^2 \times \frac{4.15 \times 10^{-9}}{n_{xF}} - 0.0071^2 \times \frac{0.077}{n_F}\right]$$

$$+ \frac{1}{n}\left[1540^2 \times \frac{2.46 \times 10^{-9}}{n_{xW}} - 0.0055^2 \times \frac{7.7}{n_W}\right]$$

$$+ \frac{10}{n}\left[0.92^2 \times \frac{6.4 \times 10^{-6}}{n_n} - 0.28^2 \times \frac{0.000.02}{n_n}\right]$$

$$+ \frac{0.23}{n^2}$$

$$V(e_x) = \frac{1}{n}\left[\frac{0.00098}{n_{xF}} + \frac{0.000039}{n_F}\right.$$

$$+ \frac{0.00584}{n_{xW}} + \frac{0.00023}{n_W}$$

$$+ \frac{0.000054}{n_{xn}} + \frac{0.00016}{n_n}$$

$$\left. + \frac{0.23}{n}\right]$$

[redacted]

[redacted]

Since the number of weighings of each feed and waste drum has been assumed for the overall material balance on T to be $n_F = 2$ and $n_W = 10$ respectively, we use the same numbers here. Further, let $n_\eta = 2$ and $n_X = 2$. The corresponding quantities in the equation above then become negligible.

$$V(e_x) = \frac{0.00098}{n}\left[\frac{1}{n_{XF}} + \frac{6}{n_{XW}} + \frac{230}{n}\right] \qquad (39)$$

$$d(0.05, e_x) = 2\sqrt{V(e_x)} = 0.063\sqrt{\frac{1}{n}\left(\frac{1}{n_{XF}} + \frac{6}{n_{XW}} + \frac{230}{n}\right)} \qquad (40)$$

This equation should be compared with Equation (28) for the material balance on T. It is interesting to note that here the most important assay is that of the waste drums, while in the T-balance the waste-weighing was more important than the feed weighing. Similarly, it does not pay here to repeat waste assays until (or unless) the material balance on X is to be run for over a month. Table VIII indicates the expected precision of the determination of daily rate of unaccounted-for loss of X for various values of n_{XF}, n_{XW}, and n, i.e., of frequency of assay of feed and waste drums and number of days between X-inventories.

[redacted]

[redacted]

TABLE VIII

Precision of Estimate of Unaccounted Loss of X, As It Depends on Number of Assays of Each Feed Drum, of Each Waste Drum, and on Number of Days between Inventories

	95% Confidence Belt on Total Unaccounted Loss of X, kgms. X		95% Confidence Belt on Daily Average Rate of Unaccounted Loss of X. kgms. X/day	
No. of Assays:				
Per Feed Drum	1	4	1	4
Per Waste Drum	6	24	6	24
Days Between Inventories				
5	1.0	1.0	0.20	0.20
10	1.0	1.0	0.10	0.10
20	1.0	1.0	0.052	0.049
30	1.1	1.0	0.036	0.033
60	1.2	1.0	0.020	0.017
90	1.3	1.1	0.014	0.012
120	1.4	1.1	0.0115	0.0090
180	1.5	1.1	0.0086	0.0063
240	1.7	1.2	0.0070	0.0050
360	2.0	1.3	0.0055	0.0036

[redacted]

[redacted]

The values given in Table VIII for $n_{XW} = 6$ and $n_{XF} = 1$ are plotted as the lower curves on Figures 3 and 4. The assumptions are summarized in Table X.

Inspection of Table VIII shows that, even for an X-balance running for 60 days, there is little to be gained by repeated assays of the main streams. Since the X-inventory term is by far the largest, it is apparent that precision in estimating X-losses can only be greatly improved by improving the precision of the X-inventory. But, except for revisions downward of some of the estimates of variances used, it does not seem likely that a great deal can be done to decrease the several variance terms shown in Table VI. The plant has a considerable amount of operational variability which could only be decreased after a much deeper study than this of the multitude of causes of variability that appear to be present.

Warning: Equation (40) and Table VIII, take account only of unaccounted-for-X due to random variability in all quantities measured. It will be shown in Section 12 that even very small, unknown biases in measurements of the X-concentration of feed and waste streams, greatly increase the overall uncertainty in Unaccounted-for-X. An equation will be given (47) taking account of all known sources of uncertainty, whether random or systematic.

[redacted]

[redacted]

12. Accuracy of X-Stream Measurements

The preceding discussion of errors in the X material balance has been concerned with random errors, whose magnitude can be estimated with reasonable accuracy, and whose effect on the material balance can be made as small as one wishes by increasing the number of measurements and/or the number of days between inventories.

In addition to these random errors, each of the quantities entering the material balance equation will be affected by consistent errors, or bias, which remain constant during the entire period of measurement. The effect of these consistent errors on the X material balance cannot be reduced by increasing the number of measurements, and they therefore set a definite, but uncertain, lower limit to error in the X material balance, no matter how many measurements are taken or how many days are allowed to elapse between inventories.

The X material balance equation which is to investigated for the effect of bias is

$$e_x = \frac{1}{\eta}\left\{\sum_{1}^{N_F} \bar{x}_F \overline{F} - \sum_{1}^{N_W} \bar{x}_W \overline{W} - \sum_{1}^{N_n} \bar{x}_n \overline{\Pi} - \Delta X_c\right\} \qquad (41)$$

The terms in this equation have been defined following equation (34)

Bias in $\overline{F}$, $\overline{W}$ and $\overline{\Pi}$ would take the form of consistent errors in scale calibrations or failure to allow for contaminants in feed, product, or waste. We shall assume that calibration

[redacted]

[redacted]

errors are negligible (less than 0.1%) and that contaminants are either absent or analysed for. It should be noted that contaminants have occasionally been reported in the product; these are not now being analysed for and constitute a possible source of error in the X material balance which should be checked.

The effect of bias in the change in X inventory has already been allowed for (Part 10), so that ΔX_c may be neglected here.

We have then to investigate the effect of bias in the feed, waste and product assays.

At the present time product is assayed by absolute measurements by the mass spectrometer. No information is available at present concerning the accuracy of these measurements, although it is unlikely that a systematic error exists larger than 1% of the X-concentration for X-concentration above 10%. It is proposed to prepare synthetically standard samples of material near product purity by mixing weighed amounts of nearly pure U-238 (about 0.03 weight per cent X) and highly enriched X. Assay of these standards by the absolute method will indicate the accuracy of the method (within the precision to which the concentration of the standard is known).

The feed assay at present is very uncertain, values being reported by different laboratories between 1 part X in 136 and 1 part X in 139. It is proposed to prepare a synthetic standard of about this concentration, and to determine the actual feed purity by mass spectrometer and fission count relative

[redacted]

[redacted]

measurements of feed against standard. In this way the feed assay can be determined very nearly to the precision with which the concentration of the standard is known.

The waste assay at present is determined by fission count relative measurements against feed. The evaluation of bias in these relative measurements is impossible at present. It is believed to be not greater than 0.5% of the ratio. The preparation of synthetic standards of about feed and waste purities will permit such an evaluation, both by fission count and mass spectrographic methods.

Equation (41) will be rewritten in terms of x_F, x_W/x_F, and x_Π, the quantities whose systematic error of measurement are thought to be most nearly independent of each other in the statistical sense.

$$e_x = x_F(\bar{F} - \frac{x_W}{x_F}\bar{W}) - x_\Pi \bar{\Pi} - \Delta X_c \qquad (41a)$$

where

$\bar{F}$ = daily feed rate in kgm. T/day

$\bar{W}$ = daily waste rate in kgm T/day

$\bar{\Pi}$ = daily product rate in kgm T/day

let $V_b(x_F)$, $V_v(x_W/x_F)$, and $V_b(x_\Pi)$ represent the variance due to bias in the assay quantities. (For example, the value of $V_b(x_F)$ would be estimated from the precision of a synthetic standard used for a careful comparison with normal feed.) The resulting variance $V_b(e_x)$ due to bias is given by

[redacted]

[redacted]

$$V_b(e_x) = \left(\frac{F x_F - W x_W}{x_F}\right)^2 V_b(x_F) + W^2 x_F^2 V_b\left(\frac{x_W}{x_F}\right) + \Pi^2 V_b(x_\Pi) \qquad (42)$$

now, $F x_F - W x_W \doteq \Pi x_\Pi$ and

$$V_b(x_F) = \left[\frac{d(0.05, x_F)}{2}\right]^2 = \left(\frac{p_1 x_F}{200}\right)^2$$

$$V_b\left(\frac{x_W}{x_F}\right) = \left[\frac{d(0.05, x_W/x_F)}{2}\right]^2 = \left(\frac{p_2 \cdot x_W}{200 \cdot x_F}\right)^2$$

$$V_b(x_\Pi) = \left[\frac{d(0.05, x_\Pi)}{2}\right]^2 = \left(\frac{p_3}{200} x_\Pi\right)^2$$

$$\therefore V_b(e_x) = \left[\frac{\Pi x_\Pi}{200} p_1\right]^2 + \left[\frac{W x_W}{200} p\right]^2 + \left[\frac{\Pi x_\Pi}{200} p_3\right]^2 \qquad (43)$$

where p_1 = 95% confidence belt in x_F, expressed as a percent of x_F

p_2 = 95% confidence belt in x_W/x_F expressed as a percent of x_W/x_F

p_3 = 95% confidence belt in x expressed as a percent of x

We now assume numerical values for the quantities in Equation (43). Πx_Π is about 2.7 kgm. $W x_W$ will be taken as 6.2 kgm.

$$V_b(e_x) = 0.000182\left[p_1^2 + 5.3 p_2^2 + p_3^2\right] \qquad (44)$$

or $$d_b(0.05, e_x) = 0.027\sqrt{p_1^2 + 5.3 p_2^2 + p_3^2} \qquad (45)$$

[redacted]

[redacted]

It will be worth while to examine the value of d_b for some probable values of the ρ:

	Assumed Percentage Bias: Feed Assay, x_F	Ratio of Waste to Feed Assay, x_W/x_F	Product Assay x_η	Uncertainty in Daily rate of Unaccounted Loss of X Due to Bias in Assay $d_b(0.05, e_X)$	Number of Days in Inventory Period Such That Uncertainty in Daily Rate of Unaccounted Loss of X due to Bias in Assays Just Equals That Due to Random Errors
I	1.5	0.5	1.0	0.058 Kgm/day	17
II	0.7	0.3	0.7	0.033 " "	35
III	0.23	0.1	0.23	0.011 " "	120

The first line corresponds to the writers' best estimate of current conditions (3/18/46). It will be seen that the uncertainty due to biases in x_F, x_W/x_F, and x_η amounts to 58 gms. per day. This is about the same as the uncertainty due to random errors, for a material balance run for 17 days. By equating these two uncertainties the maximum improvement in precision (of e_X) per day of waiting is gained. For shorter periods the random uncertainty plays an increasingly larger part. For longer periods the systematic error prevents a corresponding gain in overall precision.

It will be noted that the uncertainty in x_W/x_F is the most important in that it is more heavily weighted in Equation (45). The second and third lines in the table make the contribution of each of the three uncertainties the same. This last

[redacted]

[redacted]

line represents conditions which it is hoped will be reached within the next few months at K-25

13. Total Uncertainty of X-Material Balance

Equation (44) will now be combined with Equation (39) to give an equation permitting the estimation of the total uncertainty of a material balance on X. (V_t represents the total variance due to random errors and bias.)

$$V_t(e_x) = \frac{0.000[illegible]}{n}\left[\frac{[illegible]}{n_{xr}} + \frac{[illegible]}{n_{xn}} + \frac{430}{n}\right]$$
$$+ 0.000[illegible]\left[p_1^2 + 5.3p_2^2 + p_3^2\right]$$

Let $n_{xr} = 1$ and $n_{xn} = 6$, since larger values will not greatly decrease $V_t(e_x)$.

$$V_t(e_x) \doteq 0.002\left\{\frac{1}{n}\left[1 + \frac{115}{n}\right] + 0.1\left[p_1^2 + 5.3p_2^2 + p_3^2\right]\right\} \quad (46)$$

$$\text{or} \quad d_t(0.05, e_x) = 0.09\sqrt{\frac{1}{n}\left[1 + \frac{115}{n}\right] + 0.1\left[p_1^2 + 5.3p_2^2 + p_3^2\right]} \quad (47)$$

This equation is graphed in Figure 4 for three sets of possible values of p_1, p_2 and p_3. Since p_2 is the decisive bias, it is given values of 0.5, 0.3 and 0.1. The other two per cent biases are given such values that they make the same contribution to the total uncertainty in e_x. The total uncertainty in an X-balance run over n days is shown in Figure 3.

[redacted]

[redacted]

It is seen that, under present conditions of bias, an uncertainty of 30 kgms X/year is possible, even if all other requirements of this report are met. If, however, the conditions of "Assumption III" can be met, then not more than ± 5.8 kgms. X per year will be lost without being detected.

14. <u>Precision Required for Miscellaneous Minor Streams and Inventories.</u>

The above analysis has ignored a number of minor streams crossing the main process material balance envelope, and minor inventories within the envelope. Most of the streams are small and are normally determined with sufficient precision that they make no noticeable contribution to uncertainty in the material balance. In this category are

Laboratory Samples (20)

TF_6 lost from alumina and carbon traps (26 and 27)

TF_6 from fluorination of converters (42)

T Oxides from diffusional equipment, vacuum pumps and oil, and miscellaneous materials (52, 62, and 72)

Enriched TF_6 cascade feed (14)

Miscellaneous cascade withdrawals (16)

The following streams should be measured with precisions set according to criteria similar to those used in setting the precision of normal feed and waste:

Depleted TF_6 Cascade Feed (13)

Waste from mobile unit (19)

[redacted]

[redacted]

The precision required for spent alumina and carbon (34 and 30) will be discussed below.

The inventories of the following operations should be reduced to zero at inventory time or evaluated by post-inventory cleanout.

- Mobile UF_6 Removal Unit (I)
- Section 630 Waste Accumulator (H)
- Purge and Recovery Vacuum Pumps (L)
- Purge and Recovery Cold Traps (M)
- Fluorination of Converters (Q)
- Decontamination Operation (R, T, and V)
- Recovery Operations (S, V and W)

It would be desirable to dump all aluminum and carbon traps (J and N) at each inventory time, thus reducing their T-inventories to zero. However, there is little loss of precision in the overall material balance if some traps remain undumped for a number of inventory periods, provided their X-content is known from their operating history to be less than a specified amount. On the assumption that 24 carbon and aluminum traps are normally in use, no trap need be dumped at inventory time if its X-inventory is known to be less than 0.05 kgms. An uncertainty of 0.05 kgm. X in each trap will result in a 6% increase in the uncertainty of the total X-inventory within the material balance envelope. This is a tolerable increase.

The T and X content of traps which are dumped should be determined with sufficient precision to add a negligible amount to the uncertainty of the material balance. This will not

[redacted]

[redacted]

require a prohibitive number of samples for chemical isotopic analysis, since the amount of T and X involved is usually small.

15. <u>Localization of Losses.</u>

Losses of T or X in each Section of the Cascade cannot be determined by material balance methods on T or X. For Section 4, with the smallest flow of process gas, the heads flow is about 5000 kgm. T per day. The estimated consumption of T in this section is 0.12 kgm. per day. Thus the flow into the building would have to be reliably estimated to about ten parts per million in order to measure the T consumed with ±50% precision. This ratio is entirely unrealizable and therefore a different method for localizing losses of T and X must be devised.

By making reasonable guesses about the relative consumption of T in the different sections of the cascade it is possible to estimate the average X-concentration of the consumed material. The X and T material balances will permit an independent estimate of the X-concentration of the unaccounted losses. If the latter value is significantly lower than the former, it will be apparent that a disproportionate amount of T is being lost in the lower parts of the cascade. This need cause no great concern but a rough indication can be given of which cascade Section should be investigated.

If, however, the average concentration of X lost is significantly higher than is expected on consumption-grounds, then the probability is high that material is being lost from the upper parts of the plant. If it seems likely that the

[redacted]

[redacted]

disproportionate loss is in Section 4, then the likelihood of illegal diversion is greater, and a very careful investigation would have to be made to rule out this possibility.

16. Material Balance on U-234.

A balance on U-234 (Z) can be set up for the same envelope used in the T and X balances. Assays by the alpha-counting method are more precise than either the fission-counting or the spectrometric method for X. It will not be worthwhile to carry through the calculations for the precision of such a balance at this time, but it is recommended that a single full set of cascade inventory samples be taken and analyzed for Z so that an estimate of the possible precision can be made.

If the distribution of Z turns out to be roughly the same as that for X, then the Z balance can be used as an independent check on overall unaccounted loss of T and X. If, however, the distribution of Z is more heavily weighted toward the top of the cascade, then the material balance on Z will be more sensitive to withdrawals, losses, or diversions from Section 4, and hence will provide valuable control data.

[redacted]

[redacted]

VIII RECOMMENDATIONS CONCERNING MATERIAL ACCOUNTING PROCEDURES

1. Introduction

The primary object of the Process Development Department in preparing this report has been to analyse the problems encountered in accounting for T and X in the diffusion plant, so that specific procedures for material accounting can be set up by the Operating Departments who will be directly responsible for this work. These specific procedures will be determined in part by the considerations of this report, and in part by the practical convenience and expense of putting a particular procedure into effect. For this reason, the report does not make detailed recommendations concerning specific procedures. Nevertheless, there are certain features that seem to be so desirable in any material accounting system that they will be stated in this Section of the report, under the following headings:

General Technical Principles

Organization of Material Accounting

Notes on Specific Procedures

Notes on Further Technical Studies

2. General Technical Principles

(a) Steps in a Complete Measurement

In determining the amount of T and X held up in a particular operation or transferred from one operation to another, four steps are essential to a complete measurement:

(1) Measurement of the weight or volume of material involved.

(2) Taking a representative sample.

[redacted]

[redacted]

(3) Determination of the T content of the sample, to be reported as weight % T in the case of material whose weight is known, or as concentration of T (weight of T per unit volume) in the case of material whose volume is known.

(4) Determination of the X assay of the T present in the sample.

An adequate material accounting procedure will either direct that all four of these steps be carried out or give assumptions which may be used to provide the equivalent information. Three examples will serve to illustrate this rule:

Example (1) Complete Determination of T and X in Spent Carbon

The net weight of the contents of a drum of spent carbon is determined. A representative sample of the drum contents is taken by approved sampling methods. The T content and the X assay of the sample are determined by the laboratory. This procedure makes use of all four steps. However, if the carbon is known to have been used for the absorption of TF_6 from normal feed drums exclusively, the X assay of the sample may be omitted and a value assigned from the assay of feed reported for the period during which the carbon was in use.

Example (2) T Analysis Assumed for Cascade Waste

The net weight of the contents of a waste drum is determined. The contents are melted and agitated, and a representative sample of the liquid is run off. The X assay of this sample is determined. On the evidence of line recorder analyses for the period during which the waste was collected, its T content is

[redacted]

assumed to be equivalent to 100% TF_6. Thus, T analysis of the waste sample may be omitted.

Example (3) No Sample or Analysis of Cascade Feed

In this example it will be assumed that a drum of TF_6 feed has been sampled and analysed when originally received by Coded Chemicals. The drum is reweighed when it is shipped from Coded Chemicals to Process and is weighed again when it is returned partially emptied by Process. If the handling of the drum has been such as precludes the possibility of contamination of the drum, no samples need be taken or analyses made when the drum is shipped to Process or returned by it. The assumption is made that the original sample and analytical results properly describe the T and X content of the materials withdrawn by Process.

(b) General Points Concerning Inventory Determinations

One of the most important precautions to be observed in taking inventory is that all materials containing T are inventoried once and that none are inventoried more than once. One important aid to this precaution is simultaneous identification of the material to be inventoried throughout the entire plant. All personnel concerned should be informed of the exact time to which the inventory is to refer, with enough advance notice to be prepared to carry out their duties at that time.

Inventory times mark the beginning and end of material balance periods. These periods should be of approximately equal lengths; an interval of four weeks or one month between inventories has been suggested as appropriate in the diffusion plant.

[redacted]

Two general procedures may be used at inventory time. On the one hand, material whose flow cannot be interrupted must be measured and sampled precisely at inventory time. For instance, the cascade inventory must be determined by simultaneously recording all tails pressures, valve angles and nitrogen concentrations, and taking assay samples as close as possible to the time to which the inventory is to refer. If these operations are not carried out simultaneously, a surge of material may occur from one part of the plant inventoried before the surge to another part of the plant inventoried after it, and a fraction of the T may be inventoried twice.

On the other hand, material which is or can be isolated may be set aside so that T cannot be added to it or removed from it, and the amount of T and X determined at the operator's convenience. For instance, the inventory of a particular waste accumulator could be determined by valving off the accumulator at inventory time and later draining, weighing and sampling its contents.

Three methods available for taking inventory of operations through which flow may be interrupted at inventory times are:

(1) Direct measurement of inventory

(2) Pre-inventory clean out

(3) Post-inventory clean out

Direct measurement is the obvious procedure of weighing the contents of a drum or measuring the liquid level or pressure in a vessel, and taking a representative sample for analysis.

When it is impossible to measure the amount of material held up in an operation with sufficient accuracy (as in a cold trap),

[redacted]

[redacted]

or when a representative sample cannot be secured (as in a carbon trap), the second or third procedure must be resorted to. Of these, the second is preferable. It is simply the obvious expedient of draining or emptying the vessel just before inventory time, so that at that moment it does not contain a significant amount of T or X. As an example of this procedure, all cold traps should be empty at inventory time.

When it is impossible to measure inventory directly or when all T-containing materials cannot be cleaned out of an operation at inventory time, the third method, or post-inventory clean-out, must be used. In this, all material isolated at inventory time is processed through subsequent operations without loss of identity until it is converted to such a form that it can be accurately measured and a representative sample secured. Draining a waste accumulator to determine the weight of TF_6 in it is a simple illustration of this procedure. A more complicated example of this method is afforded by the procedure recommended for taking inventory of decontamination and recovery operations, which will be described in the following paragraphs.

The particular example of taking inventory of T and X in operations R and S, decontamination of and recovery of T and X from diffusional equipment, is cited. It will be assumed that at inventory time there will be in process in these operations a number of pump parts to be decontaminated, solutions and sludge in the decontamination tanks, and solutions and precipitates in the recovery equipment. Because of the heterogenous character of this material, it will be difficult or impossible to determine

[redacted]

the amount of T and X in it at inventory time. The material in the decontamination tanks consists of a mixture of sludge, scale and solution which will be difficult to sample in a representative manner, and the pump parts contain a layer of reduced T whose amount obviously cannot be determined without further processing.

The first step in taking inventory of these assorted materials is to identify them, and all forms into which they may be converted, in some distinctive manner, so that they cannot possibly be mixed or confused with other material to be subsequently put through these operations. The use of tags whose color differs from the tags used on material to be processed in the next material accounting period, to identify all pieces of equipment and all operations in which T is to be recovered and credited to this inventory, is an expedient found useful at Y-12. A second step is to defer processing material from the next material accounting period until all of the material to be inventoried has been put through a particular operation. For instance, no diffusional equipment from the next period would be decontaminated until all of the pump parts on hand at inventory time had been decontaminated and the solutions and sludges in the decontaminating tanks completely transferred to the recovery department. The final step is to process all of the material to be inventoried into readily weighed and sampled T oxide. The T and X found in this oxide is credited to the inventory of operations R and S at inventory time. In the procedure as described, no effort is made to distinguish between the inventory of the individual operations. If desired, this could be done by cleaning out S first, and then cleaning out R and S altogether.

Post-inventory cleanout has disadvantages:

(1) The results may be low if losses occur during processing.

(2) The results are delayed until processing is complete.

(3) The use of the operation for the next period is deferred until it is no longer required to complete the inventory.

Despite these disadvantages, post-inventory cleanout is occasionally the only method of taking inventory which can be used. Nevertheless, it is obviously preferable to clean out equipment prior to inventory time and refrain from using it until after that time has passed, whenever possible.

When an accurate material accounting system is to be put into effect, it is particularly important that all miscellaneous materials containing T and X either be cleaned out before the first period starts, accurately measured in the first inventory or carefully segregated from subsequent processing until they are converted to inventoriable form and charged as an additional shipment to the plant.

(c) General Points Concerning Transfers

To determine the amount of material which has been transferred from one operation to another it is preferable whenever possible to weigh or measure the volume of batches of material transferred, instead of measuring the flow rate and integrating it to obtain the total quantity of material. Similarly, it is preferable to take a representative sample of batches of material transferred than to take a continuous sample at a rate proportional to the rate of transfer. An exception may be made when it is known that the composition of the stream is substantially constant.

It is desirable that two independent measurements be made of the amount of material transferred at the most important points, to catch an occasional accidental error, such as misreading of a scale. These two measurements can advantageously be made by the operation shipping the material and by the one receiving it. The material accounting procedure should specify which measurement is the official one, or whether the average should be used. Ordinarily, one sample at the point of origin or receipt should suffice.

When the weight of material is evaluated from the difference in weights between a full and empty container, these two weights should always be determined on the same scales.

The irregular, unlistable, unpredictable transfers and changes in inventory that may occur in a plant as large as K-25 constitutes a serious potential source of error, because a single one of them may completely vitiate the estimate of consumption that is the primary goal of the material balances.

A crew of engineers will have to be charged with responsibility of seeing that no "spare drums" happen to be lying about, or are removed from the material balance envelope during the balance period. Borrowings for experimental purposes, drainings from large pieces of equipment, spills in Section 600, vented process gas in Section 312, and the like are all likely to be hidden or-

[redacted]

forgotten unless their importance is recognized and a procedure for reporting such incidents is established.

3. Organization of Material Accounting

The most important single factor in ensuring the success of a material accounting system is proper organization of personnel and methods for the work. Material accounting can be simplified and made more accurate if carried out according to a systematic plan and schedule, and if all persons involved are working under the same set of instructions. Since these subjects are concerned with plant organization rather than with technical procedures, a discussion of them is perhaps out of place in a technical report. Nevertheless, they are so important to successful material balances that this part of the report has been devoted to general suggestions for systematizing material accounting, which should be of value in any specific plan or organization developed for this purpose.

(a) Material Accounting Section

A material accounting section should be organized, with a technical staff composed of chemical engineers and accountants. This section should consult frequently and work closely with the Analytical Laboratory and the Process Development Department.

(b) Material Accounting Office

Headquarters of this Section should be located in a material accounting office from which material accounting procedures should be issued, to which the results of material accounting measurements by the operating departments and the Laboratory should be forwarded and from which reports on plant inventory,

[redacted]

yields and material balances should be distributed.

(c) Flow Sheet and Terminology

Under the leadership of the Material Accounting Section, a material flow sheet and terminology for describing material handling should be approved and adopted by the Material Accounting Section, the Process Division, and the Plant Management. Figure A of this report is suggested as a point of departure for this purpose.

(d) Detailed Material Accounting Procedures.

The Material Accounting Section should draft detailed procedures for accounting for material in every operation and every transfer represented in the approved Material Flow Sheet. The responsibility for carrying out each procedure should be unambiguously assigned to a specific department. Agreement concerning all details of each procedure should be secured between the Material Accounting Section and the department to which it is assigned. It should be the responsibility of the Material Accounting Section to see that all procedures in use by the various departments are generally consistent and susceptible of giving the desired precision in overall material balances, even though some latitude in the procedures is permitted the individual departments.

(e) Inventory

The Material Accounting Section should prepare forms which will have space for all measurements required for each inventory and distribute these forms to the departments responsible for taking inventory of each operation.

[redacted]

Forms for taking inventory of connected process vessels should list every vessel for which data must be supplied. These forms should indicate what data are required for each vessel and should indicate which vessels are to be sampled. The operator taking inventory should supply all of the data requested on the form and should supply the number assigned to each sample taken for analysis.

In taking inventory of the contents of disconnected vessels in storage, the usual precautions in taking physical inventory should be observed to ensure that every container is inventoried once and none more than once. The operator should record on the data sheet the container number, its gross and tare weight or other measurements of quantity, and the number assigned the sample, if one is taken.

(f) Transfer

All transfers of material from one department to another (except laboratory samples which are described in the next paragraph) should be assigned a shipment number, preferably keyed to a master flowsheet like Figure A. Each container should be given a tag whose color is descriptive of the material balance period; on each tag should be noted the shipment number, the time of shipment, the transfer route and the number of the analytical sample, if one is taken. The department originating the shipment should send a notice of shipment to the department receiving it, with a copy to the Material Accounting Office. Similarly, the department receiving the shipment should send a receipt for it to the department originating the shipment, with a copy to the Material Accounting Office. When the material accounting procedure

[redacted]

calls for weighing or measuring a shipment, the department responsible for this operation should record the results of the measurements on its notice of shipment or receipt. When the material accounting procedure calls for taking a sample for analysis, the number and weight of the sample should be recorded on the notice of shipment or receipt. This procedure will give the Material Accounting Office all the information it needs concerning the shipment of material from one department and its receipt by another.

(g) Analytical Samples

Each sample for analysis should be given a tag whose color identifies the period in which the material sampled belongs. The data to be noted on the tag will depend on whether the sample represents an inventory or a transfer, as noted in the following table:

Inventory	Transfer
Operation Letter	Stream Number
Description of Operation	Description of Stream
Batch, Lot, Vessel, or Drum Number (if any)	Shipment Number
Sample Number	Sample Number
Date of Sample	Date of Sample
Time of Sample	Time of Sample
Weight of Sample	Weight of Sample

(h) Request for Analysis

Each sample sent to the laboratory for analysis should be accompanied by a request for analysis which should specify the results desired from the laboratory and the method of analysis or

[redacted]

degree of precision required. A copy of each request for analysis should be sent to the Material Accounting Office. Each request for analysis should contain all of the information given on the sample tag.

(i) Reports of Analysis

After laboratory has completed a group of analyses, these should be reported to the department submitting the samples and to the Material Accounting Office. The reports of analysis should contain enough of the information listed in the request for analysis to positively identify the sample and avoid confusion with any other material.

(j) Scheduling Analytical Results

Since it will ordinarily be impossible to work up a complete material balance until all analytical results have been reported, it will be important for the laboratory and Material Accounting Section to develop a procedure which will prevent unnecessary delay in the completion of the samples from a particular material accounting period.

(k) Units

The units in which all data are to be reported to the Material Accounting Office should be agreed on in advance. Complete consistency in reporting is apparently not obtainable because, for example, some scales are calibrated in pounds while others are calibrated in grams. Reports from the Material Accounting Section should be consistently in one set of units, preferably metric.

[redacted]

(1) Codes

In the past, the use of codes to disguise the true concentration figures led to occasional errors. It would be highly desirable if permission could be secured from the Army to dispense with codes altogether. If this cannot be done, the code should be simple, like the one now in use, and if it is necessary to change it, the change should be made only at the beginning of a new material accounting period.

4. Notes on Specific Procedures

Before final specific procedures can be established for each of the operations and transfers shown in Figure A, a careful study of each item by the Material Accounting Section and the responsible operating department, with consultation by the Engineering Department, will be needed. Provisional procedures should be drafted, given a trial, and modified as required to improve precision or simplify operations. Study of some of the more important specific procedures has been initiated by the Engineering Department. Notes on specific procedures recommended for each transfer are summarized in Table IX and those for each inventory in Table X. These notes recapitulate recommendations made at various points throughout the text.

When procedures call for repeated weighings to improve precision, it is of the utmost importance that the weighings be statistically independent. This requirement can be closely met if:

a. Different operators make the different weighings.

b. The several weighings are made on each drum at different times of day, requiring the removal and replacing of each drum on the scales.

c. No operator is allowed to see the weight recorded by another operator.

d. Some adaptation of the new "statistical weighing" techniques is made. (This problem will be studied.)

Table IX calls for 10 independent weighings of each full waste drum. It will be especially important in this case that the drum be removed from the scales, replaced and reweighed by skilled and conscientious operators who understand the importance of their results.

5. Further Technical Studies

Further technical studies which will be helpful in developing these specific procedures have been given in Section III.

NOTES ON SPECIFIC PROCEDURES RECOMMENDED [illegible]

TABLE II

Transfer	Title	Notes on Procedure
1	Receipts of Normal TF_6	Weigh drums twice to 1%. [illegible] samples submitted by Harshaw with each lot of feed fo[illegible] [illegible]. Make vapor phase sample of each drum for assay (one per drum).
2	Shipments of Feed Cylinder Residues	Weigh drums twice on same scales used for (1).
3	Receipts of Depleted TF_6	Weigh drums twice to 1%. For this or (13), melt and agitate contents, sample, assay [illegible] analyze for T.
4	Receipts of Enriched TF_6	One weighing of drum will suffice. Melt and agitate contents, sample, assay for X and [illegible] for T.
5	Product Shipments	For preliminary records use weight of cylinder and assay of product stream determined [illegible]. For official records, use data obtained after contents of cylinder or batch of cylinders have been di[illegible] in measured weight of water at Y-12. Samples of [illegible] solution are to be returned to K-25 for T analysis [illegible] assay.
6	Miscellaneous Shipments	Depends on amount of material shipped.
~	Non-volatile T receipts	Depends on amount and nature of material. Should be weighed, sampled and analyzed for [illegible]
11	Normal TF_6 Cascade Feed	Weigh drums twice on same scales used for (1). See note on (1).
12	TF_6 Converted at K-25 area	Weigh drums once. Melt and agitate contents, sample and analyze, unless samples have [illegible] taken at (91), (92) or (93).
13	Depleted TF_6 Cascade Feed	Weigh drums twice before charging and twice after charging on same scales used for (3) [illegible] analysis reported under (3) or (C).
14	Enriched TF_6 Cascade Feed	Weigh drums once. Melt and agitate contents, sample and analyze, unless sample has al[illegible] been taken at (4), (12), (91), (92), or (93).
15	Cascade product	See (5).
16	Miscellaneous cascade withdrawals	Exact procedure depends on amount of material.
17	Non-volatile T	Unless identical with batch weighed and analyzed under (7), should be weighed, sampled [illegible] analyzed for T and X.
18	Waste from Section 630	Make ten independent weighings of [illegible] waste drums and two independent weighings of em[illegible] drums, all on same scales, to 1%. Melt and agitate drum contents, sample liquid phase, analyze for [illegible] 6 assays for X.
19	Waste from mobile unit	Section (18).
20	Laboratory Samples	Estimate or weigh samples with sufficient precision that total weight of T shipped to [illegible] laboratory is known to within 100 gm/month.
20A	K-27 Product to storage	Weigh drums twice when full and twice when empty, to 1%. Sample vapor phase and make [illegible] on each drum.
20B	K-25 Waste to storage	See (18).
21	TF_6 Fed to Purge and Recovery Vacuum Pumps.	No data required.

(Continued)

[redacted]

Table (Continued)

Transfer	Title	Notes on Procedure
22	TF_6 Fed to Cold Traps	No data required.
23	TF_6 Returned to Cascade from Cold Traps	No data required.
24	TF_6 Fed to Carbon Traps	Keep rough estimate of total cubic feet of G-74 transferred.
25	TF_6 Fed to Alumina Traps	Ditto.
26	TF_6 Lost from Alumina Traps	Estimate T content of gases by trace indicator or space recorder.
27	TF_6 Lost from Carbon Traps	Ditto.
28	Condensed waste	No reliable measurement possible.
29	TF_6 to Mobile Unit	No reliable measurement possible.
30	Spent Carbon	Take representative sample of each trap, analyze for T and assay for X. Weigh contents of each drum.
31	Spent Carbon to On-Area Conversion	Use data from (30) if available. If not, take representative sample of each lot, analyze for T and assay for X. Weigh contents of each drum.
32	Spent Carbon to Dead storage	See (31).
33	Spent Carbon to Off-Area Recovery	See (31)
34	Spent Alumina	See (30).
40	Contaminated Converters to be Fluorinated.	Keep record of serial number, location in plant and period of service of converters.
41	TF_6 Discharged with Conditioning Gases	No direct measurement possible. Estimate from increase in inventory of (Z) [illegible]
42	TF_6 from Converters	Weigh and assay.
43	Fluorinated Converters for re-use	Keep record of serial number of converters and where installed in plant.
44	Scrapped Converters	Keep record of serial number of converters. Prove that X content is less than specified weight by alpha activity inspection.
45	Fluorinated Converters to be Decontaminated	See (43).
50	Contaminated Diffusional Equipment	Keep record of type, serial number, location in plant and period of service of all equipment.

(Continued)

TABLE [illegible] (Continued)

Transfer	Title	Notes on Procedure
51	Decontamination Solutions from Diffusional Equipment	Unless it can be proved that representative samples of those solutions can be taken, no measurements are necessary.
52	T oxides from Diffusional Equipment	Weigh, sample, analyze for T and assay for X.
53	Decontaminated Diffusional Equipment	Keep record of type, serial number and where installed in plant.
54	Scrapped Diffusional Equipment	Keep record of type and serial number. Prove that X content is less than specified weight by alpha activity inspection.
60	Contaminated Vacuum Pumps and Oil	See (50). Measure oil.
61	Decontamination Solutions from Vacuum Pumps and Oil	See (51).
62	T Oxides from Vacuum Pumps and Oil	See (52).
63	Decontaminated Vacuum Pumps and Oil	See (53). Measure oil.
64	Scrapped Vacuum Pumps and Oil	See (54). Measure oil.
70	Contaminated miscellaneous materials	Describe nature and location in plant.
71	Decontamination Solutions from Miscellaneous Materials	See (51).
72	T Oxides from Miscellaneous Materials	See (52).
80	Contaminated Laboratory wastes and Equipment	Describe nature.
81	Decontamination Solutions from Laboratory wastes and Equipment	See (51).
82	T Oxides from Laboratory Wastes	See (52).
91	TF_6 Recovered from T Oxides	Weigh, sample, analyze for T, assay for X.
92	TF_6 from Spent Carbon	Ditto.
93	TF_6 from Non-volatile T Receipts	Ditto.

TABLE X

Operation	Title	Type of Inventory Recommended	Notes on Procedure
A	Storage of normal TF_6	Direct	See (1) and (11)
B	Waste Storage	Direct	All drums on hand at first inventory should be melted, agitated and the liquid sampled and analyzed for T and X. Thereafter, drum contents may be determined in transfers (18) and (19), weighed to ½%.
C	Storage of Depleted TF_6	Direct	All drums on hand at first inventory should be weighed to ½, melted, agitated, and the liquid sampled and analyzed for T. and X. Thereafter, drum contents may be determined in transfer (3).
D	Storage of Enriched TF_6	Direct	All drums on hand at first inventory should be weighed to ½, melted, agitated, sampled and analyzed for T and X. Thereafter, drum contents may be determined in transfer (4).
E	Product Storage	Pre-inventory clean out	At present the only accurate method known for analyzing a representative sample of product is to ship to Y-12 for solution in water and analysis as noted under (5). It is thus preferable that all product inventory be shipped promptly to Y-12 so that material balance is not delayed unnecessarily by waiting for analysis of this inventory.
F	Storage of miscellaneous materials	Direct	Depends on amount on hand.
G	Storage of non-volatile T receipts	Direct	See (7).
H	Section 630 waste accumulator	Post-inventory clean out	See (18).
I	Mobile TF_6 Removal Unit	Pre-inventory clean out	Drain TF_6 and purge unit with nitrogen before inventory.
J	Alumina Traps	See remarks	Unless trap inventory is known to be less than 0.05 kgm. isolate trap at inventory time, dump and take representative sample of contents.
K	Sections 300, 400, 600, & 630 Main and Purge Cascades and Purge Systems	Direct	This is the most important inventory of the plant. Exactly at inventory time readings are to be taken of the number of onstream cells, the tails pressure, control valve angle and temperature of each cell, the frequency and the nitrogen content of each building; and a sample of TF_6 is to be taken from each cell. These and other miscellaneous readings are to be taken on forms to be drafted by the Engineering Department and worked up by procedures to be specified by this Department.
L	Purge and Recovery Vacuum Pumps	Pre-inventory clean out	Inventory of purge vacuum pumps may be assumed zero. Recovery operations should be stopped before inventory time, and all pumps purged with nitrogen.
M	Purge and Recovery cold traps	Pre-inventory clean-out	Vaporize TF_6 to Cascade and purge with nitrogen before inventory time.
N	Carbon Traps	Same as (J)	See (J).
O	Laboratory	Direct	This inventory can be simplified by cleaning out as many sample tubes and waste solutions as possible before inventory time.
P	Storage of spent Carbon and Alumina	Direct	Procedures for taking representative sample from storage drums should be investigated.

(Continued)

[redacted]

[redacted]

Operation	Title	Type of Inventory Recommended	Notes on Procedure
C, R, S, T, U, V, W, X, &Y.	All inventory of Decontamination and Recovery Departments	Post-inventory clean out	See text of Section VIII - 2 (b).
Z	C-216 Disposal Tower, K-1405	Direct	This tower should be drained and cleaned out before accurate material accounting is started. If it is later to be used for absorption of TF_6, tower and drums should be calibrated for volume and charged with sodium carbonate solution instead of sodium hydroxide, so that T will remain in solution and reliable inventories can be secured.
AA, BB, and CC.	All inventory of Conversion Department	Post-inventory clean out	This Department should be treated like the Decontamination and Recovery Departments.

[redacted]

M. Benedict
AMS

M. Benedict

C. Daniel

C. Daniel

A.M. Squires

A. M. Squires

[redacted]

APPENDIX A

Derivation of Certain Statistical and Propagation-of-Error Equations

1. Random Error.

The essence of the [illegible] of average values lies in the belief that many small disturbing factors occur at random and that, therefore, the effect of repeating a measurement is to increase the likelihood that the effects of random disturbances will balance [illegible]. We need, then, a measure of the magnitude of [illegible] disturbances of a set of measurements, and we also need a method for calculating the effect on a derived quantity, of random disturbances in a primary, or measured quantity.

The fundamental measure of scatter due to random disturbance is called the *variance*. The variance of a set of numbers is defined as the sum of the squares of the differences between each number and the arithmetic average (or mean), divided by one less than the number of numbers in the set. For a discrete set of numbers, x_i, then,

$$V(x_i) = \frac{\sum^{N} (x_i - \bar{x})^2}{N-1} \qquad \text{Def. (A-1)}$$

and for a continuous set,

$$V(x) = \int_{-\infty}^{+\infty} (x-\bar{x})^2 \, \phi(x) \, dx \qquad \text{Def. (A-2)}$$

where (x) is a "distribution Function" giving the relative frequency of occurence of x's in each infinitesimal range dx, for all values of x. The distribution is normalized so that

$$\int_{-\infty}^{+\infty} \phi(x)\, dx = 1$$

The variance of an actual set of repeated and averaged measurements, called the <u>sample-variance</u>, would be $\sum_{i=1}^{N} (x-\bar{x})^2/N$. The quantity defined above is the best estimate of the "true", or "Population" variance, based on a sample of N measurements. The proof of this fact can be found in most statistics texts, e.g. in references (1), (2), (3), (4) and (5).

The only assumption made so far is that the measurements made actually sample the (infinite) population of possible values. This is equivalent to an existence-statement about the population, and to the assumption that the individual measurements are independent of each other, i.e. random. These assumptions are clearly necessary if the estimated population variance $V(x_i)$ is to be used in prediction or in description, since if the underlying population changes, or has changed during the measurements, then the set of numbers obtained has sampled nothing definite and no prediction can be made.

Expressions for the variance of sums, products, and sums of products will now be developed since all the equations describing inventories and material balances are of these forms. The important assumptions required will be indicated in the course of the derivations and then summarized in a group.

[redacted]

Suppose that a quantity A is known to be the sum of two quantities, B and C, each of which is measured separately

$$A = B + C \tag{A-3}$$

Assume that an error, ΔB, is made in measuring B and an error, ΔC, is made in measuring C. Obviously,

$$A + \Delta A = (B + \Delta B) + (C + \Delta C) \tag{A-4}$$

where ΔA is the error made in estimating A using Equation (A-3)

or
$$\Delta A = \Delta B + \Delta C \tag{A-5}$$

and
$$(\Delta A)^2 = (\Delta B)^2 + (\Delta C)^2 + 2(\Delta B)(\Delta C) \tag{A-6}$$

Now assume that B and C are each measured a large number of times, N. An equation of the form of (A-6) will hold for each pair of measurements. These equations may be added to give:

$$\sum_N (\Delta A)^2 = \sum_N (\Delta B)^2 + \sum_N (\Delta C)^2 + 2\sum_N (\Delta B)(\Delta C) \tag{A-7}$$

We now make our second major assumption; that of <u>statistical independence</u>. We assume that errors in measuring B are not correlated with errors in measuring C. A positive error in measuring C, as with a negative one. If this is the case, then the sum $\sum_N (\Delta B)(\Delta C)$ will not be large, but the other two terms on the right of Equation (A-7) will increase without limit as the number of repeated measurements of B and of C is increased. We may then write

$$\frac{\sum(\Delta A)^2}{N-1} = \frac{\sum(\Delta B)^2}{N-1} + \frac{\sum(\Delta C)^2}{N-1} \tag{A-8}$$

Each of the three quantities in Equation (A-8) is a variance, as can be seen by comparing this equation with Equation (A-1). The quantity on the left is an estimate of V(A), as deduced from the estimates of V(B) and V(C) which appear on the right. We may write, therefore,

$$V(A) = V(B) + V(C) \tag{A-9}$$

Expressed in words this equation simply states that, (under the assumption of statistical independence of the two summands) the variance of the sum of two terms is equal to the sum of the variances of the terms.

It is obvious that a similar rule holds for the sum of any number of independently measured terms. Thus

$$\text{If} \qquad A = \sum_i B_i \tag{A-10}$$

$$\text{Then} \quad V(A) = \sum_i V(B_i) \tag{A-11}$$

The operations of "summing" and "taking the variance of" are, then, commutative.

$$V(\sum \quad) = \sum V(\quad) \tag{A-12}$$

The analogy between the derivation of these equations and those of elementary calculus is quite close and will become clearer below.

It should be emphasized here that no assumption whatever about the *form* of the actual frequency-distribution of the errors in A, B, and C has been made. The equations derived above and all of those below are, of course, valid if the distribution of errors of measurement follows the Gaussian, or "normal" law, but this restriction is not at all necessary. Several recent books dealing with propagation-of-error equations for physical scientists are in error on this point (e.g. Sherwood, T. K. and Reed, C. E. - Applied Mathematics in Chemical Engineering, pages 375 et seq.).

Equations 2 and 4 of Section VIII are particularly valuable because they show how to estimate the uncertainty in an <u>average</u> of N independent measurements. Equation 4 can be derived directly from Equation (A-11). Suppose that all the B_i are measures with the same variance.

$$V(B_1) = V(B_2) = --- = V(B_K)$$

$$V(A) = \sum_{i=1}^{K} V(B_i) = K\, V(B_i) \qquad \text{(A-13)}$$

Since $$A = \sum B_i = K \bar{B} \qquad \text{(A-14)}$$

$$V(A) = K^2 V(\bar{B}) \qquad \text{(A-15)}$$

as can be seen by carrying through the operations symbolized by Equations (A-3) to (A-9), putting a constant factor before the B of Equation (A-3)

$$\therefore\ K^2 V(\bar{B}) = K\, V(B_i) \qquad \text{(A-16)}$$

(A-17)

$$V(\bar{B}) = V(B_i)/K \qquad (A-17)$$

and, taking the square root of both sides

$$s(\bar{B}) = \frac{s(B)}{\sqrt{K}} \qquad (A-18)$$

Changing now to the nomenclature of Equation 2,

$$s(\bar{x}) = \frac{s(x)}{\sqrt{N}} \qquad (A-19)$$

since the K of Equation (A-18) is simply the number of measurements of a single physical entity. This relation too, is independent of the form of the error-distribution-law, although it should be remembered that a large sample, i.e. a large N, has been assumed in this derivation. It may be shown by combinatorial methods, that the same equation is valid for small N.

The expression for the variance of the product of two measured quantities is somewhat different but equally simple. We place

$$A = B \times C \qquad (A-20)$$

Then find $$A + \Delta A = (B + \Delta B)(C + \Delta C)$$

or $$\frac{\Delta A}{A} = \frac{\Delta B}{B} + \frac{\Delta C}{C} + \frac{\Delta B \cdot \Delta C}{B \cdot C} \qquad (A-21)$$

The last term may be neglected when B and C are small with respect to B and C, respectively.

Squaring and summing over N repeated measurements of B and C, dividing by N - 1 and remembering that $2 \sum_N \frac{\Delta B \cdot \Delta C}{N-1}$ approaches zero, when B and C are statistically independent, we have:

[redacted]

$$\frac{1}{A^2}\sum_N \frac{(\Delta A)^2}{N-1} = \frac{1}{B^2}\sum_N \frac{(\Delta B)^2}{N-1} + \frac{1}{C^2}\sum_N \frac{(\Delta C)^2}{N-1} \qquad (A\text{-}22)$$

or
$$\frac{V(A)}{A^2} = \frac{V(B)}{B^2} + \frac{V(C)}{C^2} \qquad (A\text{-}23)$$

Generalizing for the product of more than two factors

If
$$A = B \times C \times D \times --- \qquad (A\text{-}24)$$

$$\frac{V(A)}{A^2} = \frac{V(B)}{B^2} + \frac{V(C)}{C^2} + \frac{V(D)}{D^2} + --- \qquad (A\text{-}25)$$

By analogy with Equations (A-10) and (A-11)

if
$$A = \prod^{i} B_i \qquad (A\text{-}26)$$

Then
$$\frac{V(A)}{A^2} = \sum_i \frac{V(B_i)}{B_i^2} \qquad (A\text{-}27)$$

Combining the fact expressed in Equation (A-27) with that given by Equation (A-11), we can show how the variance of a sum of products may be estimated. This is the algebraic case corresponding to an inventory (or material balance)

letting
$$A = \sum_i B_i \times C_i \times --- \qquad (A\text{-}28)$$

$$V(A) = \sum_i A_i^2 \left[\frac{V(B_i)}{B_i^2} + \frac{V(C_i)}{C_i^2} + --- \right] \qquad (A\text{-}29)$$

$$= \sum_i A_i^2 \sum_\ell \frac{V(z_{i\ell})}{z_{i\ell}^2} \qquad (A\text{-}30)$$

where $z_{i\ell}$ represents each of the measured variables B_i, C_i, etc; that is, the index ℓ runs through all variables, B, C, ---- and i runs through all products $B_i \times C_i \times$ ---- whose

[redacted]

sum is known to be equal to A.

The form of Equation (A-28) shows that Equation (A-30) is applicable to material-balance situations in which the algebraic <u>sum</u> of a number of streams and inventory-changes must be estimated. Each stream or inventory-change is found by measuring several factors, such as pressures, concentrations, temperatures, purities, etc., and these measured factors are the z_{ij} of Equation (A-30).

The relations derived above between the variances of measured quantities and the variance of derived quantities, e.g. of sums, products, and sums of products, are special cases of a general relation originally given by K. F. Gauss. His method used Taylor's series-expansion of a function in the neighborhood of its mean value. For any differentiable function of K variables, $z = f(x_1, x_2, \ldots x_K)$, one can express a small increment of z, (Δz), as:

$$\Delta z = \sum_{n=1}^{\infty} \frac{1}{n!}\left[\sum_{i=1}^{K} \Delta x_i \frac{\partial z}{\partial x_i}\right] f \qquad \text{(A-31)}$$

The sum in the brackets is taken over all K variables, and represents the sum of the products of the n-th power of a small increment in each variable by the n-th partial derivative of the function with respect to the same variable. The summation sign outside the brackets indicates that all terms of the type indicated above, for n equal to each integer from 1 to ∞, are to be added. The first (or right hand) summation, therefore, takes care of the K variables; the second summation takes care of the different orders of differential coefficients in the Taylor series.

It is clear that the three variances developed above, namely, for $Z = xy$, $Z_2 = A \cdot xy$, $Z_3 = \sum_i x_i y_i$ are special cases, all of which might well be derived from Equation (A-31). The derivations given are, however, simpler and more instructive, since the analogy with the methods of elementary calculus is made explicit.

If we assume that terms higher than first order may be dropped*, then Equation (A-31) becomes

$$\Delta Z = \sum_{i=1}^{k} \frac{\partial z}{\partial x_i} \Delta x_i \tag{A-32}$$

and the usual operations of squaring, summing over sets of measurements, and dividing by one less than the number of sets of measurements give

$$V(z) = \sum_{i=1}^{k} \left(\frac{\partial z}{\partial x_i} \right)^2 V(x_i) \tag{A-33}$$

This equation is quite general. Equations (A-9), (A-23), and (A-30) may all be derived directly from it.

---

* Higher order terms may be dropped when:

a. The x_i are "small", i.e. small enough so that their squares suffice to give negligible terms in the summation inside brackets in Equation (A-31).

b. The functional relation is not "sharply curved", i.e. the higher derivatives are small enough to make the higher order terms negligible. For functions of the form $ax_1 + bx_2 + \text{----}$, all higher derivatives are, of course, exactly zero.

[redacted]

Equation (A-30) may be put into a form that will help in judging the relative contribution to total random error of many different items.

Let $\rho(y) = \dfrac{s(y)}{y}$, which may be called the fractional standard of deviation of y

$\pi(y) = \dfrac{d(0.05, y)}{y}$, the fractional 95% confidence bolt on $\bar{y}$

and $f(y_i) = \dfrac{\bar{y}_i}{y_c}$, the fraction of cascade y (y_c) that is in vessel or location i.

We have been implicitly assuming thus far that each variable is measured but once. If some quantities are measured more than once in a given location, i, then, the $V(\bar{z}_{il})$ will be used in Equation (A-30), and these may be replaced by $V(z_{il})/n_{zil}$ where n_{zil} represents the number of repeated (but statistically independent) measurements made of the variable z_{il}, at the locus i. Making these substitutions in Equation (A-30), we have, for cascade inventory of X; (X_c)

$$\rho^2(X_c) = \sum_i f^2(X_i) \sum_l \frac{\rho^2(z_{il})}{n_{zil}} \quad \text{(A-34)}$$

$$\pi^2(X_c) = \sum_i f^2(X_i) \sum_l \frac{\pi^2(z_{il})}{n_{zil}} \quad \text{(A-35)}$$

In these two equations the summation over l (types of variable measured) may contain different numbers of terms for different vessels, or loci. The index i contributes a term to the overall sum for every vessel, or location, to which a single group of measurements, Z_{il}, is expected to apply.

These equations indicate the quantities that must be estimated for a preliminary study of the precision of the cascade's inventory of T or U (or U-234), and they also will indicate how precisely measurements must be made in different vessels for a given over-all precision.

It is clear that the major sources of random error will be those vessels containing the most inventory, since the terms of Equations (A-34) and (A-35) are weighted as the squares of the fractional inventories. Further, the major contributions to random error due to the precision of each type of measurement can be seen by the relative magnitudes of the $\beta(Z_{il})$ and the $\pi(Z_{il})$. Similarly the feasibility of cutting down the contribution of any particular variable's contribution by increasing the n_{il} can be studied.

It will be well now to summarize the assumptions made in deriving the above equations for the propagation of random errors.

1. The same set of random disturbing factors operate with the same relative frequencies on each set of measures of each kind. Thus $V(Z_{il})$ (where Z_{il}

is an observed quantity) is assumed to be the same at all inventory periods.

2. Errors of measurement of each variable are not correlated with each other.

3. Errors of measurement of one variable are not correlated with those of another.

4. The errors (or variations between measurements) must be small fractions of the quantities measured.

5. A sufficient number of independent measurements of each type have been made so that the used in Equation (A-30) are not seriously in error.

It will be noted that the equations used for numerical estimates in this report are all of the form of Equations (A-30), (A-34) and (A-35).

2. <u>Systematic Error.</u>

A standard practice recommended to physical scientists and to engineers is that of estimating the maximum possible error in a derived numerical result due to the maximum possible systematic errors in several constituent subsidiary observed quantities. The recommendation almost always takes the form:

$$\delta(y) = \sum_{i}^{n} \left| \frac{\partial y}{\partial x_i} \delta x_i \right| \tag{A-36}$$

where $\delta(y)$ is described as the maximum possible systematic error in $y = y(x_1, x_2 \cdots x_i \cdots x_n)$

$\delta(x_i)$ is given the value of the maximum possible systematic error in x_i

and the absolute values are summed.

The error in using this equation arises from the ambiguous meaning of the term "maximum possible systematic error". The numbers that are inserted for the $\delta(x_i)$ are almost never the absolute maximum possible errors; they are systematic errors whose probability of occurrence is judged sufficiently small. Once this apparently small qualification is accepted, however, the basis for Equation (A-36) is removed. Take as a simple example the systematic error in a sum A, of two measured quantities, B and C. Let us suppose that a systematic error of unknown sign but of magnitude 1 is thought to be "possible" in both B and C. If "possible" is interpreted to mean "of probability p", where p is small (say 0.01), then it is clear that the probability of a systematic error of magnitude ± 2 in A is 0.01^2 or 0.0001. Similarly, if there were r terms whose sum was A, each of which had a possible (e.g. 0.01 probable) systematic error of ± 1, then the probability of an error in A of magnitude r, would be p^r. Thus Equation (A-36) requires a highly variable shift in the meaning of the word possible as it is applied to $\delta(x_i)$ and $\delta(y)$. Systematic errors (of unknown sign) in $\delta(x_i)$ that are quite possible, will produce errors of the magnitude given by Equation (A-36), with an extreme rareness, and the form of the equation gives no clue as to how rare this might be.

The criticism summarized above gives an indication of one way out of the difficulty, however. If a systematic error, or bias, is thought to be 0.01 probable then it is possible to form an estimate of the "variance of bias" and the manner of combining variances has already been explained. The difference between estimates of random error, and estimates of bias is that in the former case, a single set of statistically independent measurements is all that is required, while for the latter case, different systems of measurement should be sampled. Thus for random error variance, we need only sample a single universe of measurements while for systematic error, one should obtain a random sample of systems. Evidence on random variance is given by data taken within the system (or method) of measurement being used. Such measurements give no information at all concerning bias or systematic error of the type under discussion.

It may happen that all measurements are taken on a particular variable by the same method. Say, however, that it is suspected that this method may be systematically in error by 1 unit. If the term "may be" means that a probability of 0.01 is a fair estimate of the likelihood of a bias of ±1 unit, then an equally fair estimate of the variance of bias will be

$$\frac{1}{t^2(0.05, n)} = \frac{1}{2.6^2} = 0.15$$

and this variance may be combined with other suspected biases by Equations (A-30), (A-34) or (A-35). If the "Variance of Bias" of the desired derived quantity is not negligible with respect to the variance of random error, then the two should be added to give a total estimate of uncertainty.

The derivation of Equations 46 and 47 in Section VII of this report provide examples of this situation. If, as is now the case at K-25, the ratio of U-238 to U-235 in normal feed material is thought to lie somewhere between 136 and 139, then an average value, properly weighted if need be, should be used, and a confidence belt on bias should be computed with a specified level of certainty. It would be fair at present to say that we are "95% certain" that the true value of this ratio lies in the range 137.5 ± 1.5. From this assumption, the estimated variance of bias would be $\left(\frac{1.5}{2}\right)^2 = 0.56$.

~~SECRET~~

BIBLIOGRAPHY

(1) Croxton, F. E., and Cowden, D. J., - Applied General Statistics, Prentice Hall

(2) Deming, W. E., - Statistical Adjustment of Data, Wiley and Sons

(3) Freeman, H. A., - Industrial Statistics - Wiley and Sons

(4) Rider, P. R., - An Introduction to Modern Statistical Methods - Wiley and Sons

(5) Wilks, S. S. - Mathematical Statistics - Princeton University Press

[redacted]

APPENDIX B

Detailed Calculation of Errors in T-Inventory

The variance in T inventory of Section 300 cells, caused by random errors in the five variable list below are:

	Variable	Variance
(a)	Control valve angle	53.1
(b)	Temperature	1.3
(c)	Per cent nitrogen	0.03
(d)	Per cent C-816	2.0
(e)	Per cent oxygen	< 0.03
	Total	56.5 $(kgmT)^2$

The following paragraphs describe the estimation of these variance.

The influence of control valve angle on stage inventory may be expressed by an equation of the form:

$$T_{ik} = T_{io} \left[1 - b_i \left(CV_{ik} - CV_{io} \right) \right] \qquad (B\text{-}1)$$

where T_{ik} = inventory of k th stage of size i at mean control valve position CV_{ik},% closed

T_{io} = inventory of stage of size i at nominal control valve angle CV_{io}

b_i = constant characteristic of i th size of equipment

The variance of the T inventory of the kth stage of the i th size is given by:

$$V(T_{ik}) = \left[1 - b_i \left(CV_{ik} - CV_{io} \right) \right]^2 V(T_{io}) \qquad (B\text{-}2)$$
$$- T^2_{io}\, b^2_i\, V(CV_{ik})$$

[redacted]

[redacted]

Since the term in brackets is approximately unity, and since the variance of the control valve angle $V(CV_{ik})$ is constant from stage to stage of the i th size, this equation may be simplified to:

$$V(T_{ik}) = V(T_{io}) + T_{io}^2 b_i^2 V(\overline{CV}_i) \qquad \text{(B-3)}$$

Since

$$T_c = \sum_{\substack{i \\ \text{all size}}} \; \sum_{\substack{k \\ \text{all stages} \\ \text{of i th size}}} T_{ik}$$

$$V(T_c) = \sum_i \sum_k V(T_{ik}) = V(T_o) + \sum_i T_i^2 b_i^2 \frac{V(CV_i)}{n_i} \qquad \text{(B-4)}$$

where $V(T_o)$ = variance of inventory from all causes except variance in control valve angle,

and

n_i = number of stage of i th size

T_i = Total T inventory of all stages of i th size at nominal control valve angle

Exactly analogous expressions apply for (b) changes in temperature, (c) changes in per cent nitrogen, (d) changes in C-816 concentration and (e) changes in oxygen concrentration. Since the complete equation for T inventory used in this report is:

$$T_c = \sum_{i=-3}^{4} A_i n_i \overline{P}_i \left[1 - b_i(\Delta CV)\right]\left[\quad \overline{R}\right]\left[1 - d_i(\% N_2)\right] \times \qquad \text{(B-5)}$$

$$\times \left[1 - l_i(\% 816)\right]\left[1 - q_i(\% O_2)\right]$$

[redacted]

[redacted]

The corresponding variance equation is:

$$V(T_c) = \sum A_i^{o2} n_i V(P_i) + T_i^2 \sum_{r=1} V_{ir}^2 \frac{V(\ell r)}{n_{ir}} \qquad \text{(B-6)}$$

where the first term is the expression for the effect of variance in pressure on inventory given in equation (13), and where the remaining terms have the following significance:

ℓr represents each of the respective variables, control valve angle, temperature, % N_2, % C-816 and % O_2, in turn

$V_{ir} = b_i$, , d_i l_i and q_i, in turn.

n_{ir} = number of measurements of variable r made made in section i at inventory time

Table XI lists the assumptions that have been made as to the magnitudes of each of the quantities required by the right hand side of Equation B-6 above. Each section of the table refers to one of the five variables.

[redacted]

[redacted]

Table XI, Part (a)

Influence of Variance of Control-Valve Angle on Variance

of Cascade T-Inventory

$V(T_c)$ due to uncertainty in CV angle $= \sum_{i=-3}^{4} T_i^2 b_i^2 \frac{V(CV}{n_i r}$

Section	T-Inventory of Section, T_i kgms.	Coefficient Connecting Inventory for CV Deviation, b_ix103*	Number of Control Valve Readings in Section, n_{ir}	Variance in Crontrol Valve Reading V(CV)**	Variance T-Invento of Sectic kgm^2
-3		13.75	54	6.25	.446
-2		12.80	126	6.25	5.050
-1		8.89	90	6.25	6.820
1		8.89	222	6.25	15.650
2a		12.80	276	6.25	11.730
2b		12.80	588	6.25	11.270
3a		10.00	288	6.25	.761
3b		10.00	708	6.25	.723
4		13.00	576	6.25	.628
				Total	53.138

* Values of b_i are taken from "Plant Inventory as a Function of Control Valve Position & Nitrogen Concentration", by F. Zenz and E. Welsh, 12/3/45.

** This value of V(CV) is equivalent to the assumption that 95% of the control valve angle readings in each section are with $\pm 2\sqrt{6.25} = \pm 5$ units (per cent closed) of the actual control valve positions.

[redacted]

[redacted]

Table XI, Part (b)

Influence of Variance of Temperature Measurements on

Variance of Stage Cascade T-Inventory

$V(T_c)$ due to uncertainty in temperature measuements $= \sum_{i=-3}^{4} \frac{T_i^2 \; V(R)}{n_{i\sigma}}$

Section	T-inventory of Section, T_i kgms	Number of Temperature Measurments in Section, $n_{i\sigma}$	Variance in Temperature Measurement, V(R)*	Variance in T-Inventory of Section, kgm^2.T
-3		54	6.25	0.01
-2		126	6.25	.08
-1		90	6.25	.24
1		222	6.25	.55
2a		276	6.25	.20
2b		588	6.25	.19
3a		288	6.25	.02
3b		708	6.25	.02
4		576	6.25	.00
			Total	1.31

* This value of V(R) is equivalent to the assumption that 95% of the stage temperature readings in each section are within $\pm 2\sqrt{6.25} = \pm 5°F.$ of the actual temperatures.

[redacted]

[redacted]

Table XI, Part (c)

Influence of Variance of Nitrogen Concentration

Measurements on Variance of Cascade T-Inventory

$$V(T_c) \text{ due to uncertainty in } \% N_2 = \sum_{i=-3}^{4} T_i^2 d_i^2 \frac{V(\% N_2)}{n_{i\sigma}} \quad *$$

$$= 0.0035 \sum_{i=-3}^{4} T_i^2 d_i^2 \frac{(\% N_2)^2}{n_{i\sigma}}$$

Section	T-Inventory of Section, T_i kgms.	Coefficient Connecting Inventory for $\% N_2$, $d_i \times 10^3$**	Number of Readings of $\% N_2$ in Section, $n_{i\sigma}$	Mean $\% N_2$ in Section***	Variance in T-Inventory of Section kgm.^{2} T
-3		5.48	2	0.30	.0001
-2		5.00	6	0.13	.0002
-1		6.78	6	0.12	.0005
-1		6.78	10	0.18	.0037
2a		5.00	10	0.29	.0023
2b		5.00	20	0.67	.0127
3a		5.48	10	1.30	.0063
3b		5.48	24	.80	.0023
4		5.81	14	4.13	.0049
				Total	.0330

* It is assumed that 90% of the N_2 readings are within 10% of the actual N_2 concentrations. Hence

** Values of d_i are taken from memorandum of F. Zenz referred to in Part (a) of this Table.

*** These are the monthly averages for November, 1945.

[redacted]

[redacted]

Table XI, Part, (d)

Influence of Variance of Oxygen Concentration Measurements

on Variance of Cascade T-Inventory

$$V(T_c) \text{ due to uncertainty in } \%816 = \sum_{i=-3}^{4} \frac{T_i^2}{(100)^2} \frac{V(\%816)}{n_{ir}} \quad *$$

$$= 0.35 \times 10^{-6} \sum T_i^2 \frac{(\%816)^2}{n_{ir}}$$

Section	T-Inventory of Section, T, kgms	Mean %816 in Section**	Number of Readings of %816 in Section, n_{ir}	Variance of T-Inventory of Section, kgm.^{2}T
-3,-2,-1		0		0.0
1		1.25	2	1.9
2a		0.75	5	.124
2b		0.15	10	.005
3a		0		0.0
3b		0.40	12	0.003
4		0.65	7	0.0007
			Total	2.0

* It is assumed that 90% of the 816 readings are within 10% of the actual 816 concentrations Hence

$$V(\%816) = \left[\frac{0.10x\ (\%816)}{1.7}\right]^2 = 0.0035\ (\%816), \text{ since } t(0.10,20) =$$

** These are the monthly averages for December, 1945.

[redacted]

[redacted]

Table XI, Part (e)

Influence of Variance of Oxygen Concentration Measurements on Variance of Cascade T-Inventory

Since the oxygen concentration in the cascade is always much less than the nitrogen concentration, it is clear that uncertainty in its amount will affect the T-inventory uncertainty ever less than does the nitrogen concentration. Its numerical estimation is therefore omitted.

[redacted]

[redacted]

APPENDIX C

T-Inventory Uncertainty Due to Random Errors in Piping and Minor Vessel Inventory

In this appendix, intercell piping, interbuilding piping, and Sections 600 and 312 are considered.

Intercell Piping

Assuming that process gas is a perfect gas we have

$$T_{iB} = 0.0168\, N_{iB}\, \bar{P}_{iB}\, V_{iB}$$

$$\doteq 0.0168\, \frac{N_{iB}\, v_{iB}}{n_{iB}} \sum_{i=-3}^{4} \bar{P}_{iB} \qquad \text{(C-1)}$$

where T_{iB} = kgms T in cell vessels, or piping, of type B, size i

N_{iB} = number of vessels or piping units, of type B, size i

$\bar{P}_{iB}$ = average pressure, p.si.a. in cell units

v_{iB} = volume of each unit, in cubic feet

n_{iB} = number of pressure measurements made

$\bar{P}_{iB}$ = estimated average pressure in each unit, p.s.i.a.

[redacted]

The corresponding random error equation for all sizes is:

$$V(T_B) = \sum_{i=-3}^{4} \left(0.0168\, N_{iB} v_{iB}\right)^2 V\left(\bar{\bar{P}}_{iB}\right)$$

$$= \sum T_{iB}^{2} \frac{V(\bar{\bar{P}}_{iB})}{\bar{\bar{P}}_{iB}^{2}}$$

$$= \sum \frac{T_{iB}^{2}}{\bar{\bar{P}}_{iB}^{2}\, n_{iB}} V\left(\bar{P}_{iB}\right)$$

$$= \sum \frac{T_{iB}^{2}}{\bar{\bar{P}}_{iB}^{2}\, n_{iB}} \left(\frac{0.1}{2}\right)^2$$

$$= 0.71 \times 10^{-6} \sum \frac{N_{iB}^{2}\, v_{iB}^{2}}{n_{iB}} \tag{C-2}$$

We have assumed that 95% of all recorded pressure are within $\pm$ 0.1 p.s.i.a. of their mean. If, now $n_{iB} = N_{iB}$

$$V(T_B) = 0.71 \times 10^{-6} \sum n_{iB} v_{iB}^{2} \tag{C-3}$$

Numerical values are substituted in this equation in Table XII(a)
The total contribution to T_c due to intercell piping errors is 4.7 kgms^2.

[redacted]

[redacted]

Interbuilding Piping

Equation (C-3) applies to this piping unchanged. The numerical substitution is shown in part (B) of Table XII. The total amount of variance contributed to $V(T_c)$ is 1.7 $kgms^2$.

Section 600, Surge System

Considering only the parts of Section 600 that are at high pressure, since they contain almost all the T inventory, we may approximate the latter by

$$T_{600} = KV_{600} \frac{\bar{P}_{600}}{\bar{R}_{600}} \quad \text{kgms} \qquad \text{(C-4)}$$

We now assume

$\bar{P}_{600} =$ = mean pressure

$\bar{R}_{600} =$ = mean temperature

$KV_{600} = 28,200$ = Volume times a conversion factor

$T_{600} =$

The random error equation is:

$$V(T_{600}) = T_{600}^2 \left(\frac{V(\bar{P}_{600})}{\bar{P}_{600}^2} + \frac{V(\bar{R})}{\bar{R}^2} \right) \qquad \text{(C-5)}$$

[redacted]

[redacted]

If 95% of the pressures recorded can be assumed to be within 0.05 psia of the true mean pressure, then

$$V(\bar{P}_{600}) = \frac{1}{n_6}\left(\frac{.05}{2}\right)^2 \qquad \text{(C-6)}$$

$$= \frac{0.000625}{n_6}$$

where n_6 = number of independent pressure measurements averaged to give $\bar{P}_{600}$.

Similarly, if the uncertainty of measured temperature is $\pm 5°$ R, then

$$V(\bar{R}) = \frac{1}{n_7}\left(\frac{5}{2}\right)^2 = \frac{6.25}{n_7} \qquad \text{(C-7)}$$

where n_7 = number of independent temperature readings.

If, now, as is ordinarily the case, only one pressure and one temperature are recorded, then

(C-8)

$V(T_{600}) =$

$=$

$= 9.8\ (\text{kgm})^2$

[redacted]

[redacted]

Section 312 - Purge Cascade

The contribution to T-inventory uncertainty of the purge cascade inventory is negligible, being roughly estimated at 0.005.

$$T_{312} = \sum_{i=1}^{3} T_i = \sum A_i n_i \bar{\bar{P}}_i \qquad \text{(C-9)}$$

$$V(T_{312}) = \sum V(T_i) = \sum T_i^2 \frac{V(\bar{\bar{P}}_i)}{\bar{\bar{P}}_i^2} = \sum A_i^2 n_i V(\bar{\bar{P}}_i)$$

where A_i = size factor in kgm. T/cell/psia for Section 312

= ______

n_i = number of cells per building

= 16

$\therefore V(T_{312})$ =

= .0053 kgm^2

under the usual assumption that 95% of all pressures recorded actually are within $\pm$0.1 psia of the true pressure of TF_6 at that time.

[redacted]

Table XII, Part (a)

Influence of Random Errors in Measuring T-Inventory

of Intercell Piping

$$V(TB) = 0.71 \times 10^{-6} \sum_{i=-3}^{4} n_{iB} v_{iB}^{2}$$

where n_{iB} = number of sets of intercell piping per section, each set measured once.

v_{iB} = volume of each set of intercell piping, in cu. ft.

Section	n_{iB}	v_{iB}	$0.71 \times 10^{-6}\, n_{iB} v_{iB}^2$
-3	9	74	0.04
-2	21	160	0.39
-1	15	230	0.58
1	39	190	1.02
2a	46	150	0.74
2b	85	150	1.37
3a	46	70	0.16
3b	118	70	0.41
4	96	20	0.03
Total			4.74 kgm^2

[redacted]

Table XII, Part (b)

Influence of Random Errors in Measuring T-Inventory

of Interbuilding Piping

$$V(T_B) = 0.71 \times 10^{-6} \sum_{i=-3}^{4} n_{iB} v_{iB}^2$$

where n_{iB} = number of sets of interbuilding piping per section

v_{iB} = volume of each set, in cu. ft.

Section	n_{iB}	v_{iB}	$0.71 \times 10^{-6} n_{iB} v_{iB}^2$
-3	1	150	0.02
-2	3	300	0.19
-1	3	330	0.24
1	5	320	0.37
2a	5	270	0.26
2b	10	250	0.44
3a	5	150	0.08
3b	12	110	0.10
4	7	40	0.01
Total			1.71 kgm^2

[redacted]

[redacted]

APPENDIX D

Error Due to Uncertainty in Feed and Waste TF_6 Purity

The weight of T in the waste stream during an inventory period is given by

$$\overline{T}_w = \sum_{i=1}^{N_w} \overline{p}_i \overline{W}_i$$

where $\overline{T}_w$ = Total weight of T in Waste during inventory period

N_w = number of waste drums withdrawn

$\overline{p}_i$ = fractional purity, i.e. fraction of weighed material which is TF_6

$\overline{W}_i$ = weight of material in waste drum i in kgms. TF_6

The corresponding variance equation is

$$V(\overline{T}_w) = \sum_{i=1}^{N_w} V(\overline{p}_i \overline{W}_i)$$

$$= \sum_i [\overline{W}_i^2 V(\overline{p}_i) + \overline{p}_i^2 V(\overline{W}_i)]$$

Now, the second term in brackets is quite closely

$V(\overline{W}_i) = 0.77$ for a waste drum weighed 10 times

The first term should not be appreciably greater than the second term. Let us set the first term equal to 0.5. Then, since $\overline{W}_i = 1540$

$$0.5 = \overline{1540}^2 V(\overline{p}_i)$$

$$V(\overline{p}_i) = 0.5/\overline{1540}^2$$

$$d(0.05, \overline{p}_i) = 2\sqrt{V(\overline{p}_i)}$$

$$= \pm 0.001 \text{ or } \pm 0.1 \text{ per cent}$$

[redacted]

[redacted]

Correspondingly, if feed drums are weighed twice, p_4 for feed purity should be $\pm$ 0.2 per cent. It will be convenient to analyze only one sample from each lot of feed submitted by Harshaw. This sample should analyzed to a precision of 0.05%, since there are 15 drums in each lot. For product TF_6 purity, even an uncertainty of $\pm$ 2 per cent will not contribute a noticeable amount to the uncertainty of the T-balance.

In the overall material balance on X, the situation is less stringent for feed and waste, and more stringent for product. Using the criterion that the uncertainty contributed by uncertainty in chemical purity be not appreciably greater than that due to assay in each stream, it appears that feed TF_6 purity should be known to $\pm$ 1%, waste purity, and product TF_6 purity to $\pm$ 0.7%

[redacted]

APPENDIX E

Table XIII

Influence of Random Errors in Pressures and X-Assays on Uncertainty of X-Inventory.

See Section VII, Part 9, for derivation of Equation (32), which is recopied here:

V (XCells) = variance of X-inventory of cells of Section 300

$$= 81 \times 10^{-6} \sum_{j}^{N_B} X_j \left(\frac{5.1}{n_j P_j^2} + \frac{1}{n_{xj}} \right)$$

where j = building number

N_B = total number of buildings in cascade

X_j = X-inventory of buildings j

n_j = number of cells in building j

P_j = mean tails pressure in building j

n_{xj} = number of independent assays in building j

The following table lists the values of

$$A_j = X_j^2 \frac{5.1}{n_j P_j^2} \quad \text{and} \quad B_j = X_j^2 \frac{1}{n_{xj}}$$

for the case $n_{xj} = 2$. The table thus permits a building-wise comparison of the contribution of random errors in Pressures and X-assays to total uncertainty in X-inventory. This comparison permits one to select the buildings for more careful pressure instrument calibration and for repeated assays if it is desired to reduce the X-inventory variance in these ways.

[redacted]

Table XIII (Continued)

Building No. (j)	X_j	n_j	P_j	$\frac{5.1}{n_j P_j^2} X_j^2$	A_j	B_j	A_j+B_j	Section Total ($A_j + B_j$)
Section -3								
311-1		9						
Section -2								
310-3		8						
2		8						
1		5						5
Section -1								
309-3		3						
2		6						
1		6						16
Section 1								
301-1		8						
2		8						
3		8						
4		8						
5		5						61
Section 2a								
302-1		7						
2		10						
3		10						
4		10						
5		9						46
Section 2b								
303-1		9						
2		10						
3		10						
4		10						
5		10						
6		10						
7		10						
8		10						
9		10						
10		9						153
Section 3a								
304-1		9						
2		10						
3		10						
4		10						
5		9						49

[redacted]

[redacted]

Table XIII (Continued)

Building No (j)	X_j	n_j	P_j	$\frac{5.1}{n_j P_j^2} X_j^2$	A_j	B_j	A_j+B_j	Section Totals (A_j+B_j)
Section 3b								
305-1		9						
2		10						
3		10						
4		10						
5		10						
6		10						
7		10						
8		10						
9		10						
10		10						
11		10						
12		9						308
Section 4								
306-1		11						
2		14						
3		14						
4		14						
5		14						
6		14						
7		13						136
Totals for all Sections								778

Total Variance of X-Inventory due to Pressures

and Assays $81 \times 10^{-6} \times 778 = 0.063$ (KgmsX)^2

[redacted]

[redacted]

Table XIV

Summary Table: Influence of Random Errors in Pressure and X-Assays on Uncertainty of X-inventory

Section	Variance due to Assays	Variance due to Pressures	Total Variance of Cascade X-inventory due to Assays and Pressures
-3	0.0000	0.0000	0.0000
-2	0.0001	0.0003	4
-1	0.0004	0.0009	13
1	0.0012	0.0037	49
2a	0.0008	0.0029	37
2b	0.0040	0.0087	127
3a	0.0010	0.0029	39
3b	0.0132	0.0118	250
4	0.0052	0.0056	108
Totals	0.0258 kgm^2X	0.0370 kgm^2X	0.0628 kgm^2X

[redacted]

FLOW of T and X in K25 and K27 PLANTS

No.	DATE	No.	DATE
1	11/30/45		
2	1/16/46		

REVISIONS

3347

CARBIDE AND CARBON CHEMICALS CORPORATION

DRAWN BY: R.G.D. APP'D BY: M.B.

CHECKED BY: M.B. DATE: 11/29/45

DWG. NO. B 73

FLOW of T & X in K25 & K27 PLANTS FIGURE